Corrosion

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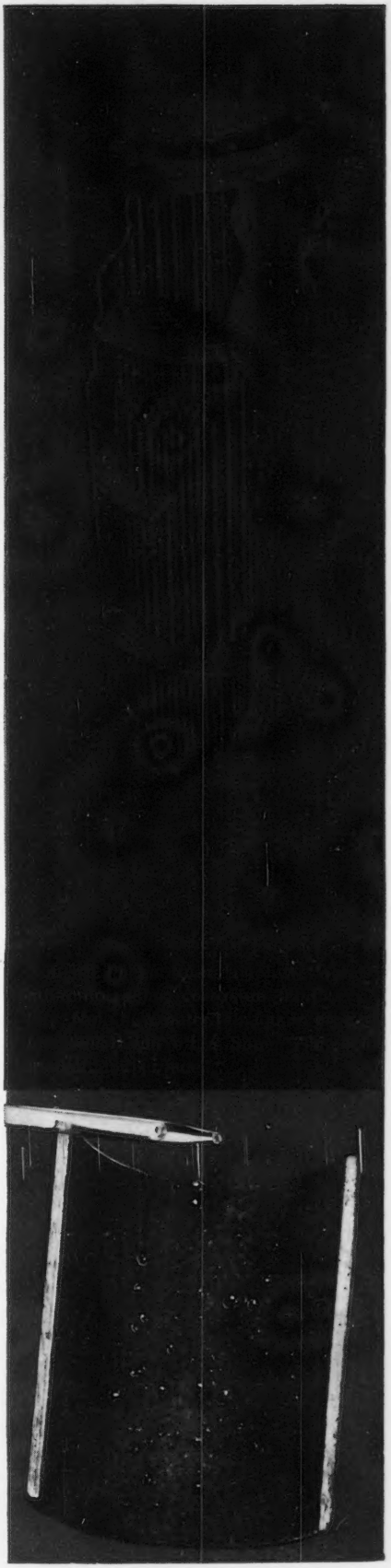

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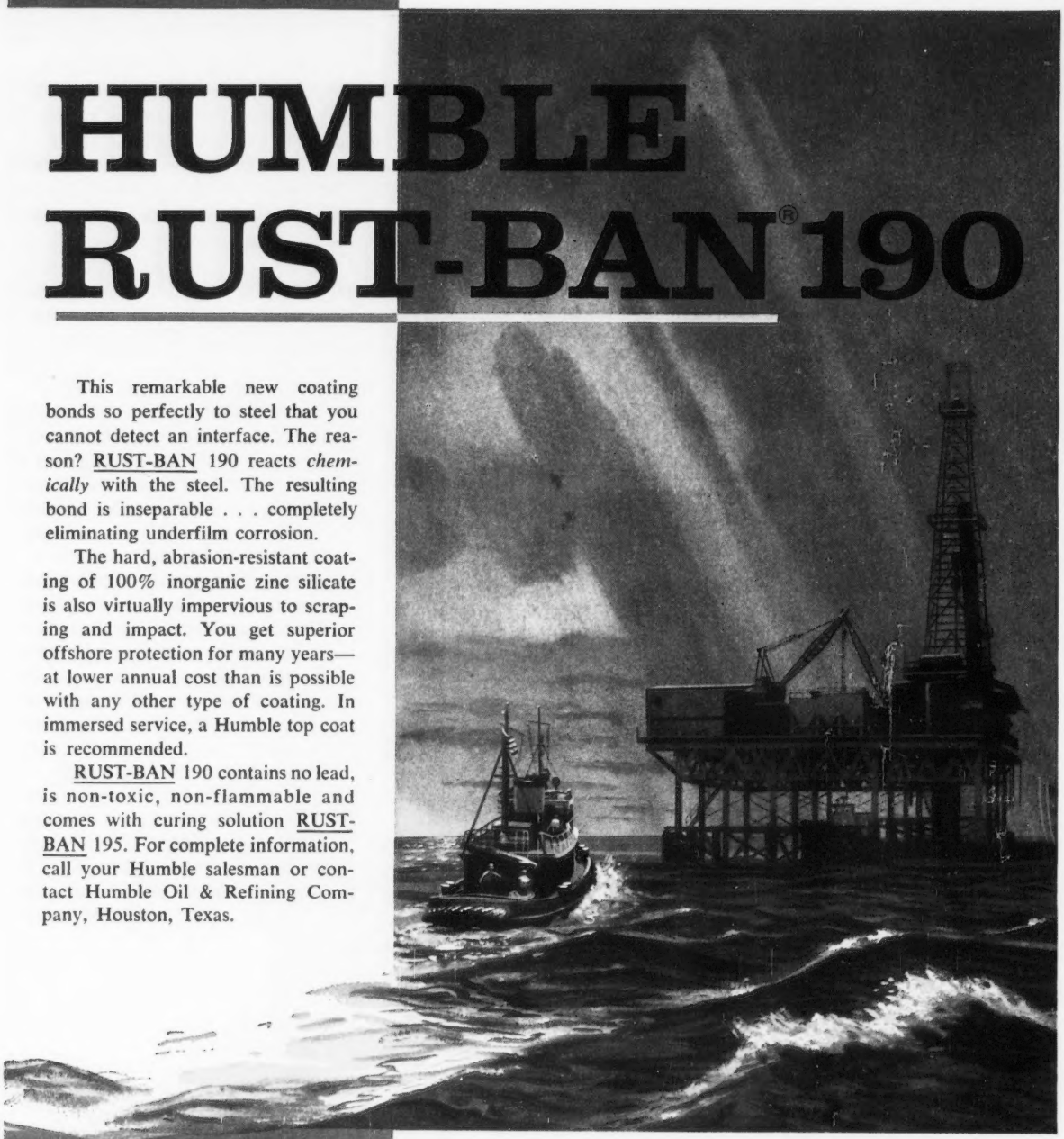

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This Month in Corrosion Control . . .

POLYAMIDE-EPOXY COATINGS have characteristics which make them different in many respects from other epoxies. Comprehensive data are given on tests of 17 primer types and performance as to wetting ability, adhesion and flexibility are reported. Turn to Page 9 for comprehensive information also on application techniques and examples of specific applications.

CRASH REPAIRS on autoclaves attacked by sodium carbonate-bicarbonate solutions involved welding of Inconel and stainless steel strips, and application of various coatings. High corrosion rates in other parts of the plant were discovered to be the consequence of a change in alkalinity. Turn to Page 20.

INSTRUMENTS TO KEEP TRACK of the water content in nitrogen tetroxide missile propellants are described in an article beginning on Page 26.

URETHANE COATINGS' characteristic differences are described beginning on Page 28. Several new types being developed are described.

REPORTS ON REGIONAL CONFERENCES in pictures and text will be found beginning on Page 35. Advance news about programs of sections and short courses also will be found.

NACE'S TWO TOP AWARDS, the Willis Rodney Whitney Award and the Frank Newman Speller Award, will be presented to two eminent corrosion experts at the 1962 Annual Conference in Kansas City, Missouri, March 19-23. Photographs and biographies of the recipients, J. E. Draley and C. P. Larrabee, appear on Page 57.

FUNDAMENTALS OF CORROSION will be the general topic of eight lectures by leading United States researchers at the 18th NACE Conference at Kansas City in March. The speakers will survey the recent advances in corrosion theory in the principal areas of interest to corrosion engineers. Turn to Page 60.

NEW OFFICERS AND DIRECTORS to be installed at the 18th Annual NACE Conference in Kansas City are President Rolland McFarland, Jr., Vice President C. G. Munger, Treasurer C. G. Gribble, Jr., and Directors W. H. Burton, Otto H. Fenner and M. E. Holmberg. Turn to Page 62 for complete story, including photographs and biographies.

STEEL CORROSION RATES in alkaline pulping digesters are materially reduced by addition of sulfur to the liquor. Turn to Page 73.

CATHODIC PROTECTION of steel pipelines in desert areas is complicated by additional environmental factors not found elsewhere. Some of the findings on systems applied to desert lines are described in an article beginning on Page 75. Among the interesting data is a 15-year record of leaks.

ZIRCONIUM surfaces prepared for exposure in high temperature water and steam will not give accurate corrosion measurements if extreme care is not observed in removing traces of hydrofluoric-nitric acid etchant. See Page 82.

INHIBITION of boiling solutions of potassium carbonate saturated with carbon dioxide and hydrogen sulfide can be accomplished with several materials. Experience with these inhibitors is described beginning on Page 87. Various combinations of phenolics and coal tar epoxies were found to be resistant to these solutions.

OXIDATION OF ZIRCONIUM in carbon dioxide at high temperatures is explored in an article from researchers in England. Turn to Page 91.

CONDENSER TUBES' reactions to river water are given in considerable detail in an article beginning on Page 95. Variables examined include effects of caustic addition, continuous cathodic protection, river water velocity, pH of water, chloride ion content and hardness, among others.

EFFECT OF HYDROGEN PICKUP and its interaction with radiation damage to zirconium in atomic reactors are among the factors bearing on materials reliability, an article beginning on Page 105 says. Data are presented also on the kinetics and phenomenology of high temperature steam corrosion.

PAINTING COSTS data derived from a survey made by NACE Technical Unit Committee T-6D are presented in a report beginning on Page 115. Among the data tabulated about painting economics in four major chemical plant categories are relationship of annual maintenance painting costs to repair outlay, capital investment and total painting; characteristics of outlay; distribution of costs for various maintenance painting activities; materials purchasing and control practices, average applied cost in cents per sq ft among others. Turn to Page 115.

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NOTICE
to readers of
CORROSION

Effective in January, contents normally appearing on this page will be found in the new NACE magazine MATERIALS PROTECTION. This will include articles listed under "Technical Topics," news of NACE activities and items formerly carried under the heading "Record and Report."

The contents listed on the facing page (5) consisting of the "Technical Section" will appear in CORROSION, to be issued as a journal for scientific and engineering articles on corrosion control about the 19th of every month.

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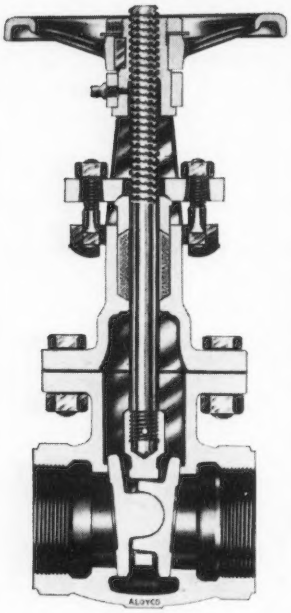

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An Expanded Program for NACE Periodicals

THIS ISSUE OF CORROSION marks the end of a significant phase in the development of the National Association of Corrosion Engineers and the ramification of its activities. It is the last issue in which news and general technical articles will be combined. It marks the end of an unbroken series of 190 issues covering a period of 17 years when CORROSION was the sole periodical issued by the association.

The new year will see NACE with three magazines instead of the present one. They will be: MATERIALS PROTECTION, a monthly devoted to the news/application aspects of NACE activities. CORROSION, a monthly journal devoted to the scientific and engineering aspects of corrosion control. CORROSION ABSTRACTS, a bi-monthly journal containing abstracts of the world's corrosion control literature.

This proliferation represents recognition by NACE that the corrosion mitigation field has developed in complexity and diversity to the point that, in the interest of good communication, the association is obliged to supply periodicals designed to meet the diverse interest and needs of its member and non-member readers. Like other technical organizations, NACE faces a communication problem of growing magnitude in carrying out its objectives. While the interest of corrosion engineers has broadened to include nearly all materials and both the fundamental as well as the so-called practical aspects of corrosion mitigation, world technology has burst into frenzied activity, creating new ideas at such a rapid rate that even the most industrious and conscientious fall behind.

In its new monthly magazine MATERIALS PROTECTION the association aims to attract the interest of and get a reasonable share of the time of its member and non-member readers. By lucidly presenting essential information about materials with which the corrosion engineer works, and by broadly covering the whole field interestingly, it expects to bring more information in a more digestible form to more people. All materials with which a corrosion worker must be familiar, as well as essential information about administration, economics, design and other significant aspects of his problems will be presented.

Likewise, in recognition of the need for continued emphasis on the more technical aspects of the corrosion worker's problems, CORROSION, as a separate entity, will be better able to concentrate on the scientific material which is the basis for real progress. With its distinguished editorial staff, this magazine expects to add to its stature as a leading authority on corrosion.

The magazine CORROSION ABSTRACTS represents NACE's effort to solve the problem of the researcher facing the formidable task of winnowing from the mass of the world's technical literature, matters significant to him. It represents the association's current thinking that a periodical is the most efficient as well as the least expensive way to make abstracts available to most of those who need them. The tested NACE Abstract Filing Index will be used and a comprehensive subject index will be provided.

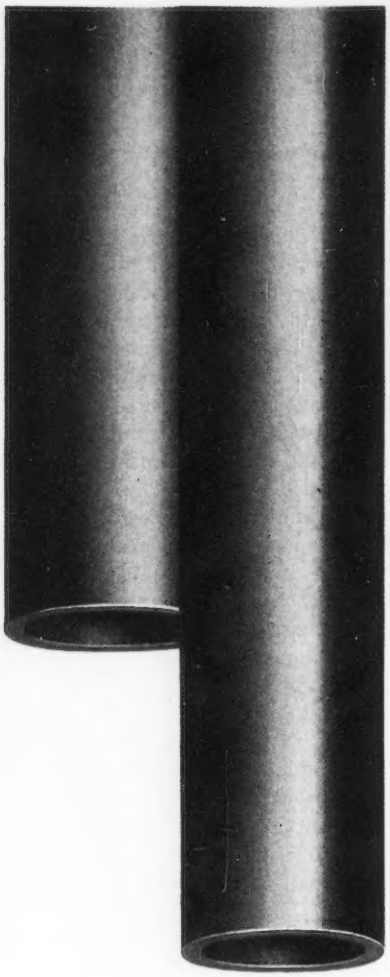
Especially in MATERIALS PROTECTION will the association seek closer identification with the needs and interest of corrosion workers. A greater volume of information on the administrative and economic aspects of the corrosion problem will be published. This is a change in emphasis from present policy of concentration on qualitative factors.

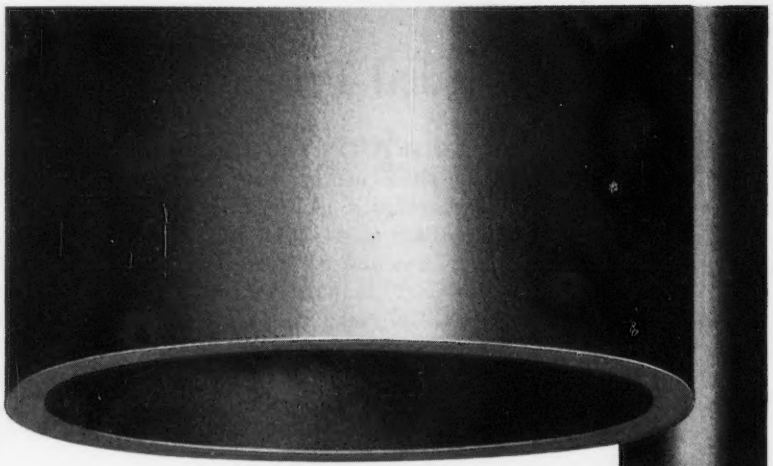
In another direction, the association hopes to explore and publicize, chiefly by means of staff articles, corrosion control problems to which the association has given but little attention heretofore. Examples of this are staff articles on automobile corrosion in this year's February and October issues.

In coming months the editors hope to find like areas of interest, many of which will be concerned with consumer goods. While these may not necessarily have urgent significance to many association members, they are rightfully a function of NACE in seeking to advance the cause of corrosion control.

Also due for more attention are technically non-corroding materials such as wood, glass and concrete. The universal application of these materials makes it necessary for the corrosion engineer to know something about them. NACE can focus attention on these materials so that they will last longer, and thus cost less.

The decision to embark on this strenuous publication program is evidence of the close attention the association's officers and board are paying to members' problems. It recognizes their diverse interests, so widely separated as to industrial interest and geography, and the wide range of their occupations. Central Office staff, implementing the board's decision, looks with confidence to improve service to the association, and faster recognition of the fact: "You *can* do something about corrosion!"





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Corrosion Control Advantages of Polyamide-Epoxy Coatings*

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Don E. Floyd
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Minneapolis, Minnesota

Abstract

Chemical and physical properties of polyamide resins are described. Resistance of General Mills' polyamide resins to various acidic and alkaline corrosives in both static and dynamic tests are compared to those achieved with commercial inhibitors. Results of tests in a semi-tropical ocean environment of 17 primer types consisting of various combinations and percentages of polyamide-epoxy vehicle with zinc, chrome and lead pigments; alkyds, phenolics, vinyls, polyvinylchlorides and a linseed oil vehicle are given. These tests of angle irons and flat panels up to 24 months showed polyamide-epoxy systems (with or without corrosion inhibiting pigments) to be better than others, even when brushed onto wet, rusty surfaces.

Wetting ability, adhesion and flexibility of the polyamide-epoxy systems as registered in several tests are reported. Also given are rate of transmission of water and water vapor.

Data are provided on formulations and application techniques. These include sand-filled coating, solventless finishes (with tests versus several corrosives and solvents). Also given are descriptions of applications as pipe coatings, marine submersion service, for aircraft and concrete. 19 references.

6.8, 5.4.5

Introduction

AMINO-CONTAINING polyamide resins of low molecular weight react with epoxy resins at room temperature or at slightly elevated temperatures to provide highly corrosion resistant vehicles for primers and topcoat paints. This article presents data to indicate that the polyamide resins themselves are corrosion inhibitors with excellent wetting properties. Mineral acid inhibition tests, National Association of Corrosion Engineers corrosion tests and dynamic corrosion testing indicate that these resins are excellent corrosion inhibitors. Interfacial tension data show their good wetting properties. In addition, they adsorb selectively on siliceous surfaces, even in the presence of large amounts of water. All these data point to the high level of surface activity of the film-forming polyamide molecule.

That these basic properties are reflected in actual coatings performance is indicated by the excellent results obtained when films are subjected to extensive exposure testing under corrosive conditions, by the ease with which the coatings may be applied to surfaces, by the excellent adhesion demonstrated and by a variety of other properties which will be described.

This article also presents application and formulation data.

Chemical and Physical Properties

The polyamide resins are low molecular weight polymers formed by the condensation of di- or polyamines such as ethylene diamine or diethylene triamine, with so-called "dimer acid." Dimer acid is a mixture of dibasic and polybasic acids obtained by the polymerization of diunsaturated fatty acids such as linoleic acid. Chemistry involved in formation of the dimer acid, as well as the chemistry of interaction with di- and polyamines has been detailed in other publications.[1,2]

When the dimer acid is condensed with a diamine such as ethylene diamine, a hard resin which may have amine end groups is obtained. However, the concentration of the free amine groups in this type of resin is so low that rapid reaction does not take place when it is combined with epoxy resins. If, however, a polyamine such as diethylene triamine is used, a fluid resin results. This resin has a high concentration of free amine groups and is useful for interaction with epoxy resins. The mechanism of this interaction has been defined by Peerman, Tolberg and Floyd.[3]

There are, of course, a variety of curing agents and coreactants for epoxy resins which make them suitable for protective coatings. It is believed, however, that the polyamide coreactants, which are film-formers in their own right, are unique in contributing the following basic properties: (1) corrosion inhibition, (2) a high degree of wetting, (3) a high degree of adhesion and (4) a high degree of flexibility.

These properties, coupled with the ability of the polyamides to cure epoxy resins effectively, make possible the formulation of maintenance paints which may be applied to a variety of surfaces under adverse conditions (e.g., high humidity, temperatures as low as 45 F, slightly rusted, wet steel). These include ferrous and non-ferrous metals, wood and plastic surfaces including phenolic and fiberglass polyester laminates. The resulting films are durable and highly protective.

The polyamides permit considerable tolerance in ratio variation with epoxy resins, as opposed, for example, to aliphatic amine curing agents. Unlike the amines, they are not irritating to the skin and are believed to be non-toxic.

The excellent corrosion resistance of polyamide-epoxy paints described in detail is based on actual exposure experience. Inherent wetting ability of polyamide resins is demonstrated by surface and interfacial tension data to be discussed. Adhesion and flexibility are much more difficult to express in quantitative terms. Quantitative data are, however, presented to show the extensibility of supported films as well as their

(Continued on Page 10)

★ Revision of a paper titled "Versamid-Epoxy Coatings—Corrosion Applications," presented at the 17th Annual Conference, National Association of Corrosion Engineers, Buffalo, N. Y., March 13-17, 1961. Journal Series Paper 260, General Mills Research Laboratories.

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high impact resistance. Extensibility on a supported surface and impact resistance are, of course, a function of adhesion and flexibility.

Polyamide Resins as Corrosion Inhibitors

Polyamide resins contain fatty residues as well as amine and amide groups. Thus, they meet the criteria for materials which are surface active, monomolecular film-forming, and corrosion-inhibiting by possessing small polar "heads" which may orient at an interface and large fatty "tails" (the classical non-polar groups of surface chemistry). It is this monomolecular fatty film, presumably, which provides corrosion protection by preventing access of oxygen, water, hydrogen sulfide and other corrosive substances to metal surfaces.

Table 1 describes the materials discussed in this article and gives their designations and Table 2 presents the results of mineral acid corrosion tests, NACE corrosion tests[4] and dynamic corrosion tests under a variety of corrosive conditions with several polyamide resins, and with related materials. Included for comparison are standard corrosion inhibitors whose activities are well known.

These corrosion data demonstrate that the polyamide resins and related materials are film-forming, corrosion-inhibiting agents. Unlike most corrosion inhibiting monomolecular film-formers, however, the polyamides are polymeric. When formulated into coatings, they contribute both their corrosion inhibition qualities and structural character.

Exposure Testing of Polyamide-Epoxy Coatings

In order to study the protection given to steel by polyamide-epoxy and other coating systems under accelerated exposure conditions but in a natural environment, coated steel panels were exposed on a tidewater beach near Miami, Florida. The selected panels were in salt water part of each day and in salt air for the remainder of the time, while also under the influence of strong sunlight.

Several authorities[5-7] have shown that in maintenance coatings one key to protection of the metal lies in selection of the primer. Thus, this study was largely limited to a comparison of different types of primers and was part of an extensive exposure study of both primer and topcoat systems.[12] The primer part of a maintenance coating system is in direct contact with the metal and is the first line of defense in protecting the steel surface against corrosion.

Metal substrates chosen in this study represented typical steel forms as used in plant construction. Two types of panels were used: angle irons and flat hot-rolled 1020 type steel plates. The angle irons measured 3 by 3⅛ by 7 inches long and had a tight oxide scale and some loose rust on the surfaces. All were cut from the same piece to minimize differences in steel composition. The flat panels measured 3 by 7 by ⅛ inches and had tight oxide scales. The steel pieces were degreased with xylene and wire brushed to remove loose rust before coating. Tightly bound scale and rust were left on the panels. Ideally, it would be preferred to sandblast surfaces to bare metal, but because this frequently is impossible in maintenance coating operations, it was more practical to apply treatments as described.

A variety of vehicles and pigmentations was used to provide a broad basis for comparison of coating types. Primer systems are identified in Table 3.

Solvents used included xylene-methyl isobutyl ketone (1:1) for the epoxy resin and xylene-Cellosolve* (9:1) for the Versamid. The latter was also used for the "let-down." It was not until after this work was undertaken that data were obtained demonstrating the virtue of butanol over Cellosolve, as will be pointed out later.

* Union Carbide tradename for ethylene glycol monoethyl ether.

Experimental Procedure

Primers were applied by brushing in alternate directions with three or four coats to obtain approximately 4 mils thickness of cured primer system. One side of each angle iron was brushed wet with water before applying the first coat of primer to simulate conditions which might be found on application in damp weather. In the case of the vinyl resin formula, it was necessary to apply five

TABLE 1—Description of Materials

Designation of Material	Trademarks	Amine Numbers
Polyamide 1	Versamid 115	220
Polyamide 2	Versamid 125	320
Polyamide 3	Versamid 140	400
Polyamide 4	Versamid 100	80–93
Polyamide 5	Genamid 250	450
Polyamide 6	Genamid 310	415
Epoxy 1	Gen Epoxy 525 (1)	...
Epoxy 2	Gen Epoxy 190 (1)	...
Low Viscosity Polyamide (LVP)	XR-2000	620

Versamid, Gen Epoxy and Genamid are registered trademarks of General Mills, Inc. XR-2000 is a polyamide-like product characterized by low viscosity. Genamids are proprietary polyamide-like products, also with low viscosities.

(1) Condensation product of bisphenol A and epichlorhydrin; M.P. for Gen Epoxy 525; about 70-72 C. Gen Epoxy 190 is a liquid at room temperature.

TABLE 2—Mineral Acid Inhibition

Materials	Conc., ppm	Conditions	Temp. C	Corrosive Agents	Percent Protection or Wt. Loss
Polyamide 1	250	Static[1]	70	5% HCl	95
Polyamide 2	250	Static	70	5% HCl	97
LVP (Low Viscosity Polyamide)	250	Static[2]	88	15% HCl	0.4 lb/ft²/day
Control	250	Static	88	15% HCl	1.1 lb/ft²/day
An accepted inhibitor	250	Static	88	15% HCl	0.3 lb/ft²/day
NACE[4] Corrosion Test					
Polyamide 1	10	Static	amb	H_2S, NaCl	65
Polyamide 3	10	Static	amb	H_2S, NaCl	64
LVP	10	Static	amb	H_2S, NaCl	88
Polyamide 5	10	Static	amb	H_2S, NaCl	81
Polyamide 6	10	Static	amb	H_2S, NaCl	94
An accepted commercial inhibitor	10	Static	amb	H_2S, HaCl	93
Dynamic Corrosion Test[3]					
Polyamide 3	50	Dynamic	65.5	H_2S, NaCl	71
LVP	50	Dynamic	65.5	H_2S, NaCl	57
Polyamide 5	50	Dynamic	65.5	H_2S, NaCl	87
A good commercial inhibitor	50	Dynamic	65.5	H_2S, NaCl	88

[1] 1 x 1 inch x 16-gauge precleaned and weighed 1020 cold rolled steel coupons are suspended in 200 ml. of 5% HCl at 70C (unstirred) for 6 hours. The coupons are weighed and weight loss data calculated as compared to a control (no inhibitor).

[2] 1 x 2½ x ⅛ inch precleaned and weighed 1020 cold rolled steel coupons are suspended in 200 ml. of 15% HCl (unstirred) at 88C for 18 hours; coupons are reweighed and data presented as weight loss in pounds per square foot surface per day.

[3] ½ x 5 inch x 16-gauge 1020 cold rolled steel coupons are immersed in 900 ml. of test fluid (1 part clay treated kerosene, 7.5 parts 5% NaCl in distilled water containing 500 ppm H_2S) at 65.5C for 24 hours rotating at two revolutions per minute. Data presented, as compared to a control (no inhibitor), are calculated from weight loss.

Polyamide-Epoxy Coatings

coats in order to obtain the proper film thickness over the usual wash primer. One or two days for drying was allowed between separate coats. The panels were later checked for pinholes with a portable high frequency generator of the Tesla coil type designed for locating leaks in a glass vacuum system. Care was taken to cover or touch-up areas containing pinholes. Thus, the coated panels were essentially pinhole free as determined with this equipment.

At this stage, all coated panels were allowed to stand at room temperature for seven days to insure that all films reached a fully cured condition. They were then installed for exposure testing on the tidewater racks near Miami, Florida.

All panels were mounted on 45-degree south exposure racks, avoiding metallic contact between panels or between panels and screws or other metal assemblies to prevent corrosion due to electrolytic action between different metal pieces.

Panels were removed from the racks and examined at six-month intervals and returned to the racks to continue the test exposures.

At each examination, panels were rated in terms of uncorroded areas remaining after exposure. In this rating system, ten is perfect and zero represents corrosion of the entire surface of the steel.

TABLE 3—Primer Systems Included In This Study

A. Polyamide 1 (35) + 7 (65) zinc yellow, 32% PVC
B. Polyamide 4 (50) + 7 (50) zinc yellow, 40% PVC
C. Polyamide 4 (50) + 7 (50) zinc yellow, 32% PVC
D. Polyamide 1 (35) + 7 (65) red lead, 34% PVC
E. Phenolic resin, red lead (TTP86-IV) 41% PVC
F. Polyamide 4 - commercial primer, zinc yellow
G. Linseed oil, red lead (TTP86-I) 36% PVC
H. Alkyd - zinc yellow (JAN-P-735) 40% PVC
I. Alkyd-zinc yellow (TTP-636) 47.5% PVC
J. Vinyl-zinc yellow (MIL-P-15930A) 36.5% PVC
K. Experimental solventless Versamid-epoxy, red lead, 10% PVC
L. Polyamide 4 (50) + 7 (50) red lead, 35% PVC
M. Red oxide commercial metal primer, alkyd, fish oil
N. Epoxy 1, diethylene triamine, red lead, 35% PVC
O. Epoxy 2, diethylene triamine, strontium chromate, 22% PVC
P. Polyamide 1 (35) + 7 (65) chrome green enamel, 10% PVC
Q. Polyamide 1 (35) + 7 (65) lead silico—chromate, 36% PVC

Test Results Noted

As noted during examinations, corrosion first began at sharp edges, particularly on the angle irons, probably because stresses are concentrated there. Also, in spite of all efforts to produce uniform films, sharp edges probably had a thinner protective layer than flat surfaces. Where corrosion occurred, this was usually accompanied by blistering and even flaking of the film. Test results after 24 months are summarized in Table 4 which relates composition to corrosion resistance.

Ester Linkages Do Not Stand Up

A number of interesting observations can be made. Most of the vehicles containing ester linkages did not stand up under these severely corrosive conditions. Such vehicles include oil-based and alkyd-based systems. Generally, the epoxy systems consisting of combinations of a polyamide and epoxy resin or epoxy resin cured with amine catalyst were far more resistant. Partial explanation for this behavior may be that alkali is formed by electrolytic action in presence of salt water. Ester groupings are not resistant to alkali, and film breakdown resulting from the saponifying action of the alkali is followed quickly by corrosion.

The vinyl system was more resistant to the effects of salt water and salt spray than were the alkyd or oil-base paints, but not as resistant as the polyamide-epoxy types. The amine-cured epoxy formula containing strontium chromate pigment gave poor performance, possibly because strontium chromate may not be as effective as zinc chromate in paint systems for corrosion inhibition. It was necessary to use strontium chromate in this particular formula because zinc chromate is reactive with amine-cured epoxy coating formulas.

By and large, the angle irons and the flat steel panels gave similar results with one or two exceptions; however, corrosion generally started more quickly on the angle irons at the sharp exposed edges. Little or no difference was noted in most instances, where performance was good, between the water wetted side of the angle iron and the dry side.

Surprisingly the chrome green enamel made with the polyamide-epoxy vehicle was almost as resistant as the zinc yellow and red lead primers, indicating that the polyamide vehicle plays an important part in corrosion protection and that, in this instance, the vehicle is at least as important as the inhibitive pig-

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TABLE 5—Interfacial Tension Data at 25 C

Product	Concentration (1)	Interfacial Tension (2)
Control (Cellosolve: Xylene, 1:9)	..	18.1
Polyamide 4	1.0	3.0
	0.1	2.9
Polyamide 1	1.0	4.8
	0.1	3.6
Polyamide 2	1.0	5.6
	0.1	2.8
Polyamide 3	1.0	2.8
	0.1	3.0
LVP	1.0	4.0
	0.1	3.7
Epoxy 1	1.0	10.2
	0.1	11.1
Ethylenediamine	1.0	12.9
	0.1	17.6

(1) Percent active ingredient in Cellosolve/xylene (1:9).
(2) Distilled water versus product in Cellosolve/xylene (1:4).

TABLE 4—Tidewater Exposure Tests
(A rating of 10 indicates essentially no corrosion)

	Angle Irons						Flat Panels			
	6 Months		12 Months		Months		Months			
Primer Type*	Dry Side	Wet Side	Dry Side	Wet Side	18	24	6	12	18	24
A	10	10	9.5	9.5	9	7	10	9.5	9	9
B	10	10	9.5	9.5	9	7	10	9	9	8
C	10	10	9	9	9	8	10	9	8	7
D	10	10	9	9.5	9	6	10	9.5	9.5	9.5
E	8	8	2.5	4	0	0	9	3	0	0
F	10	10	9.5	9.5	9	7	10	9	7	4
G	9	9	6	5	3	0	9	7	4	0
H	9.5	9.5	8	8	6	0	10	9	7	6
I	9	9	6	7	6	0	10	7	5	0
J	10	9.5	9.5	8	8	5	10	9.5	9	8
K	9.5	9.5	7	9	7	..	10	6	4	0
L	9.5	9.5	8.5	9	9	7	10	5	4	0
M	9	8	3.5	4	2	0	10	3	0	0
N	9.5	9.5	7	8	7	..	10	9.5	8	6
O	8	8	1	2	0	0	10	5	2	0
P	10	10	9	9	8	6	10	8	6	4
Q	10	10	9.5	0.5	9	8	Omitted from series			

* See Table 3.

Advantages of Polyamide-Epoxy Coatings

Figure 1—The wet aggregate on the left was coated with asphalt containing 0.5 percent Versamid 140. Over 95 percent coverage resulted. The wet aggregate on the right was coated with asphalt which did not contain Versamid. Here very little coverage was observed.

(Continued From Page 11)

ment in primer systems. This suggests that a polyamide-epoxy enamel can be applied in multiple coats over bare structural steel to offer excellent service when it is not practical to prepare the surface carefully and apply a primer.

After 24 months' exposure on a tidewater beach area, the panels were removed and the test ended.

The study showed that superior performance was demonstrated by polyamide-epoxy vehicles pigmented with the customary zinc yellow or red lead inhibitory pigments. The same vehicles in enamel formulation gave almost as good protection.

Brushing Onto Wet Surface Effective

As indicated above, these tests showed that the polyamide-epoxy system may be brushed onto a wet surface. Presumably, the surface active polyamide emulsifies the water with the solvent. Brushing is the preferred means of application on wet surfaces, because it provides the shear required for emulsification.

A dramatic example of the use of this system for direct application on sweating penstocks has been recorded in the corrosion literature.[8]

Wetting, Adhesion and Flexibility

The good corrosion inhibiting properties of polyamide-epoxy coatings are a function not only of the inherent corrosion-inhibiting activity of the polyamide resins but also of their wetting, adhesive and flexibilizing properties. Interfacial tension data which are a direct indication of wetting ability are given for polyamide resins and for related materials in Table 5 with comparative data on ethylene diamine, a commonly used curing agent for epoxy resins. Data also are provided for epoxy resins.

These data show that polyamides are strongly surface active, a property not possessed by either epoxy resins or aliphatic amine curing agents such as ethylenediamine. The data explain effectiveness of polyamide systems as corrosion resistant protective coatings which may be applied over wet, mildly rusted surfaces. The data also emphasize the importance of using sufficient polyamide in the formulation to take advantage of its wetting properties.

A striking example of polyamides' ability to promote wetting and, correspondingly, adhesion is found in the ability of the material to promote adhesion of asphalt to wet rock aggregate. A standard procedure for such a test is available[9] which involves adding asphalt to wet aggregate with mixing. Normally, little adhesion is observed. On the other hand, if asphalt containing 0.5 percent Polyamide 3 is added with mixing, over 95 percent of the wet aggregate is coated. To complete the test, the coated aggregate is cured for two hours in an oven at 140 F and then immersed once again in distilled water for 16 hours. The results are indicated in Figure 1 which shows that asphalt containing polyamide adheres well to over 95 percent of the wet aggregate after curing, whereas the untreated asphalt shows little coverage.

Film Extensibility

Film extensibility data are directly related to adhesion and flexibility, both of which are difficult to measure directly. Extensibility of both free and supported films are detailed in Table 6.

Data are given for several types of formulation. First of these is based on standard ratios and is representative of the formulations widely used for maintenance and other types of paints. Another type formulation is intended primarily for improved extensibility and includes a flexibilizing epoxy resin.

Free films for these tests were subjected to direct elongation and tensile tests. Supported films were subjected to extensibility tests with an impact flexibility tester.[1]

As might be expected from a tightly crosslinked film, free film extensibility of the polyamide-epoxy standard combinations was low as compared to the free film extensibility of alkyd resins. However, the polyamide films were hard where the alkyd resins were soft as indicated by the Sward rocker hardness values. The percentage of extensibility, however, on tin plate or steel as measured by General Electric impact flexibility tester was above 60 percent for the polyamide films as well as for the long oil alkyd. Whereas this might have been expected for the soft alkyd film, it would hardly have been expected for the hard polyamide film. Indirectly, these data demonstrate the strong adhesion of the polyamide-epoxy film to the metal surface.

The flexibilized formulation in Table 6 is recommended for use where an even greater degree of extensibility is required than is obtained with the standard formulation. Elongation of the free film

(Continued on Page 14)

(1) Most of the free films were cast on tin plate and then removed by amalgamation of the tin with mercury. Film thickness was of the order of 1.0 to 1.5 mils. The polyamide-epoxy films were cured 7 days at 75 F before testing while the alkyds were baked at 300 F for 30 minutes to insure thorough cure. Films approximately 0.5-inch wide were elongated to rupture at the rate 0.5 inch per minute using the Instron Tensile Tester Model TTC, available from Instron Engineering Corp., Canton, Ohio. The impact flexibility tester is a General Electric model available from Gardner Laboratory Corp., Bethesda 14, Md. It operates on the principle of a falling weight which impacts and extends the supporting coating.

TABLE 6—Film Extensibility

Film Composition	Sward Rocker Hardness	Percent Elongation of Free Film (ASTM D882-56T)	Percent Extensibility of Film Supported on Tinplate or Steel
Polyamide 4 Epoxy 1 (50:50)	60	3.8	60+
Polyamide 1 Epoxy 1 (35:65)	60	4.7	60+
Polyamide 2 Epoxy 1 (25:75)	60	4.0	60+
Polyamide 4 Epoxy 1 Epoxy(1)	35	50.6	60+
Short Oil Alkyd (2)	26	37	20
Long Oil Alkyd (3)	16	40	60+

(1) A flexibilizing resin Epoxy X-71 available from Shell Chemical Co. Epoxy equivalent = ca 420. Epon 1009 from the same company has an epoxy equivalent of 2400-4000. The formula is based on the following vehicle: PA 4, 60; PA 7, 40; Epon X-71 10 parts by weight.
(2) Beckosol 1307. Reichold Chemicals, Inc.
(3) Aroplaz 1241. Archer-Daniels-Midland Co.

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(Continued From Page 12)

was greater even than that of the alkyd films. It is particularly useful where flexibility, extensibility and impact resistance are required at low temperatures. It has been found useful for the painting of sharp edges and other "difficult-to-coat" areas. It is of interest for the coating and decoration of metal sheets which are later to be subjected to stress in fabrication. The higher degree of extensibility, coupled with good adhesion to rubber, suggests this system as a coating for rubber and other elastomers.

Cured 4-mil films based on this formulation will pass a 90-degree bend over a 2-inch rod at —20 F when coated on ⅛-inch steel. Three-mil films on 20-gauge steel will pass a 180-degree bend over a ¼-inch rod at 0 F. These data demonstate not only the excellent flexibility of the system but also the fact that this flexibility is retained at low temperatures.

Standard Systems Also Useful

The fact must be stressed, however, that the standard systems, utilizing standard ratios of the resins, are useful also for coating metal to be fabricated and for the coating of thin plastic films, metal foils and paper. These films will readily pass a ⅛-inch mandrel bend at 75 F. They will withstand up to 170-inch pounds of reverse impact on 20-gauge steel and, as the data in Table 6 indicate, will undergo 60 percent extension if on a metal substrate.

The adhesion of the polyamide-epoxy system is excellent not only to ferrous metals but also to a long list of nonferrous metals, glass, concrete, masonry and numerous plastic films. Among the important applications for this paint system are coatings for the hard-to-coat surfaces presented by baked phenolics and by cured polyester resin-glass laminates.

Permeability to Moisture and Gases

Another important characteristic of a film which will demonstrate good corrosion resistance when formulated into a paint is the rate at which it is permeable to moisture and gases.

Data in Table 7 give average water transmission of seven types of protective coatings films as determined by the Payne Cup procedure which is similar to method described in ASTM Standards, Method D 1653-59.

These data show that the polyamide-epoxy system has the lowest rate of water transmission and that the amine-epoxy system is second.

Data of the Pittsburgh Society for Paint Technology[10] indicate also that a polyamide-epoxy film will absorb 2.10 percent water as opposed to an epoxy-amine film which absorbed 5.36 percent. This is probably a reflection of the fatty character of the polyamide resins.

Water vapor permeability also is very low as indicated by specific permeability data in Table 8, also determined by the Payne Cup procedure.

The polyamide-epoxy system in Table 7 showed low water transmission even though butyl Cellosolve was used in the formulation. This is a solvent which normally contributes to poor water resistance because there is some tendency for the hydrophilic Cellosolve to be retained in the film. Butanol-toluene (or xylene) is the preferred solvent combination for good water resistance, and the butanol content is maintained as low as possible, consistent with satisfactory solvent power.

Formulation and Application Data

Maintenance Coatings

Polyamide-epoxy systems are used widely in primers and topcoats for maintenance coatings. Adequate film thickness normally can be achieved in three coats, and application is relatively simple. Ratio of the two resinous components is not as critical as are ratios in other epoxy systems such as amine-cured formulations.

The films cure rapidly. While polyamide-epoxy systems usually can be handled within one to three hours after application, curing takes place only with difficulty at temperatures below 45 F. Low temperature curing may be improved by addition of triphenyl phosphite. Use below 45 F, however is not recommended. Curing is strictly a time-temperature relationship and thus on hot days takes place rapidly.

Suggested starting formulations for red lead primers, zinc yellow primers, zinc rich primers and a variety of topcoat enamels have been published.[11]

Of importance in maintenance as well as other applications is the good abrasion resistance of the system. Much data illustrate this point.[11,12]

Pipe Coatings

Hardness and the glossy, smooth surfaces of the polyamide-epoxy enamels, which promote fluid flow, make them logical candidates for internal coating of pipe. For coating the pipe when manufactured, formulations based on standard ratios of polyamide and epoxy resins may be used.[11] Such formulations also are used for coating pipe which has been stored in the open and whose inside surface has corroded. Although the inside surface of such pipe is normally wire-brushed, some rust usually adheres.

For "in-place" coating of buried pipe, formulations are available with solids contents of about 80 percent.[11] Here, only small amounts of fast evaporating solvents are used for as rapid dry as possible in a confined space. The coating does not sag and makes possible deposition of a thick film on the entire inner pipe surface. Pot life is, of course, shortened by the higher solids content but remains in a practical range.

Marine Coatings

Salt spray resistance is excellent as indicated by data reported by Glaser, Floyd and Wittcoff,[1] by Glaser and Floyd[12] and by Graubert.[13]

The polyamide-epoxy system is widely used where water immersion is encountered, particularly in marine applications such as outsides of barges and tankers and inside tankers. Important in obtaining the best adhesion of the system dur-

(Continued on Page 16)

TABLE 7—Rate of Water Transmission Through Coatings Films[10]

System	Rate, Grams Per Day
Alkyd-epoxy	0.0572
Urethane	0.399
Phenolic	0.0387
Vinyl	0.0261
Epoxy ester	0.0118
Epoxy-amine	0.0097
Epoxy-polyamide	0.0089

TABLE 8—Specific Water Vapor Permeability of Coatings

System	Av. Spec.(1) Permeability
Polyamide 4, Epoxy 1 (50:50)	0.28
Polyamide 1, Epoxy 1 (35:65)	0.32
LVP, Epoxy 2 (35:65)	1.197
Polyamide 5, Epoxy 2 (30:70)	0.148
Linseed oil	3.0
Pure phenolic resin, tung oil linseed oil (33:33:33)	0.53
Epoxy resin ester (EpiVar 100, Jones Dabney Co.)	0.25

(1) Specific Permeability, D, of a film is defined as $D = gt/A$ where
g = weight of water lost in milligrams in 24 hours at 100 F.
t = film thickness in mm
A = area—10 sq. cm.

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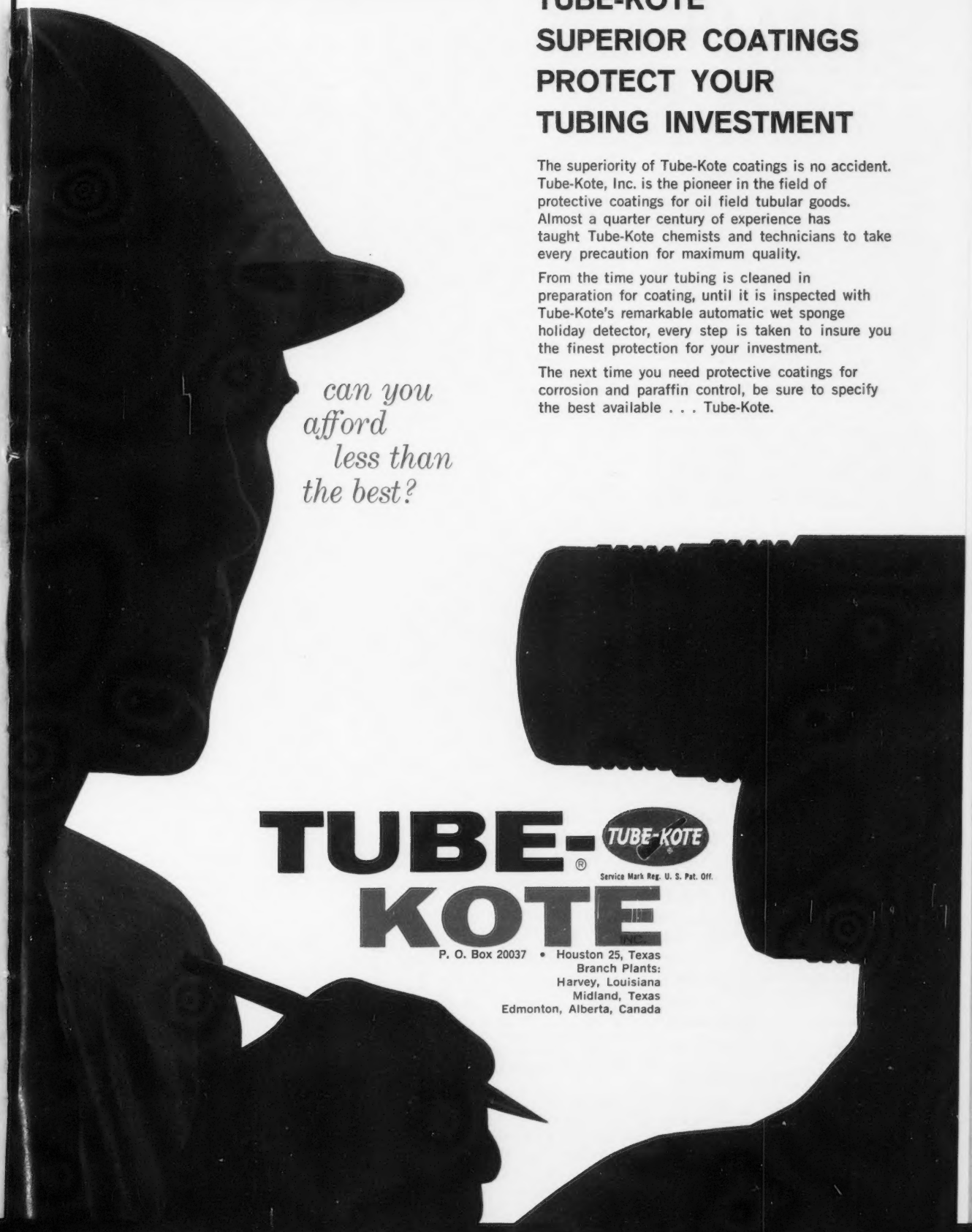

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Advantages of

(Continued From Page 14)

ing water immersion and for general marine applications are the following:

1. Formulation of coating with a minimum of alcohol-containing solvents. If alcohols must be used, butanol is preferred over Cellosolve or butyl Cellosolve.

2. Preparation of steel surface to which paint is to be applied by treatment with hot zinc acid phosphate. Aluminum can be treated with hot chromic acid to achieve the same effect. The zinc acid phosphate treatment can be conducted at room temperature for five minutes and followed, if desired, by a dilute chromic acid rinse, although this latter is not absolutely necessary unless long immersions in salt water are anticipated.

3. A vinyl butyral wash coat primer of the type described in MIL-P-15328A is effective in providing good adhesion to steel but is not effective with 24 ST aluminum. The wash coat primer is not as effective as the chemical treatments described in Paragraph 2 above.

4. An induction period of 45 to 60 minutes after mixing the polyamide and epoxy components helps contribute to good adhesion on immersion. For solventless coatings, 10 to 25 minutes is adequate.

5. Incorporation of additives such as triphenyl phosphite also helps maintain good adhesion after water immersion.

Aircraft Coatings

Good abrasion resistance, hardness and excellent adhesion to steel, aluminum and magnesium have contributed to the use of polyamide-epoxy systems for aircraft coatings. The ability to withstand rain erosion also is outstanding.

Because modern aircraft finishes frequently must be resistant to jet lubricant fluids, some modification of the standard formulation may be required. Particularly is this necessary to achieve resistance to Skydrol[2] and other aircraft lubricants. One possibility is to replace up to 50 percent of the Polyamide 1 normally used in such formulations with Polyamide 3 or LVP, both of which contribute to better solvent resistance.

Of particular interest for aircraft use is a formulation intended to protect magnesium surfaces at 500 F. This system was developed under contract to the Wright Air Development Center and has been described by Glaser and Bromstead.[14] The important part of the formulation is the use of calcium chromate with Polyamide 1 and a high molecular weight epoxy resin in the primer. The same resins comprise the topcoat vehicle which is pigmented with aluminum. This coating was judged best of many tested for protection of magnesium surfaces at high temperatures. In particular, it did not lose gloss, toughness or adhesion on exposure. At the same time, it had excellent resistance to high humidity, salt spray and jet fuel. (This formulation is described by Military Specification MIL-P-2808.)

[2] Proprietary aircraft lubricant made by Monsanto Chemical Co., St. Louis, Mo.

TABLE 9—Typical Base Formula for Sand-Filled Coating

Materials	Parts By Weight
DMP-30[1]	2
LVP	20
Epoxy 2	72
Sand	600

[1] DMP-30, Rohm & Haas Co., Philadelphia. Chemical name is Tris (dimethylaminomethyl) phenol.

Sand-filled Coatings

Sand-filled coatings are an important variation of the solventless coatings described below. Several such formulations have been described in detail.[15]

Resinous toppings for concrete and concrete patching compounds, particularly those based on combinations of LVP and epoxy resins, can be modified with coal tar cuts, asphalts or low-cost hydrocarbon polymers. This will reduce costs without seriously hurting rate-of-cure, hardness, or chemical resistance unless too much modifier is added. LVP is more tolerant of high extender contents than many other coreactants for epoxy resins.

To a typical base formula such as in Table 9, one can add as much as 94 parts of a hydrocarbon polymer extender such as "Transphalt" and 600 parts more of sand, thus doubling the size of the batch at little additional cost. Such a modified formula requires about two days to reach a firmness needed for heavy traffic. The unmodified type (without the extender or added sand) will be firm after an overnight cure. However, the hydrocarbon polymer type is equivalent to the Table 9 formulation in resistance to water, 5 percent acetic acid, JP-4 jet fuel, or gasoline.

Similar results can be expected with various coal tar and asphalt extenders if care is taken to select low viscosity grades compatible with liquid epoxy resins.

Although lesser amounts of the extender can be used if desired, increasing the ratio of extender to epoxy resin beyond that indicated here is apt to reduce hardness and chemical resistance.

Concrete topping and patching compounds, based on Polyamide 5/epoxy resin combinations also can be modified with the same extenders although somewhat less should be used. As a rule, a suggested starting point is the addition of 60 parts hydrocarbon polymer extender to 100 parts of Polyamide 5/epoxy resin blend. Adding the extender to the vehicle or binder permits increasing the sand content.

Solventless Coatings

Formulation of solventless coatings based on Polyamides 2 and 3 have been described, but Polyamide 5 and LVP both have much lower viscosities and can be handled more readily.

For example, vehicles consisting of combinations of Polyamide 5 or LVP with liquid epoxy resins such as Epoxy 2 as shown in Table 10, can be considered starting points in development of solventless finishes for a variety of applications.

When properly applied and cured, coatings based on such vehicles are resistant to chemicals and are hard and tough. Because they contain no solvent, thick films can be applied in one coat. For example, films to 10 mils thickness can be applied in one coat providing a non-sag agent such as amorphous silica is included in the formula.

Results of chemical resistance tests on 5-mil films applied to mild steel rods are described in Table 11. The coated rods were half immersed in chemicals in enclosed containers.

Also, 5 percent salt spray resistance tests at approximately 95 F with both types of formulas have given excellent results with essentially no undercutting of the film after 400 hours.

Both suggested formulas will cure satisfactorily at room temperature or at elevated temperatures. The LVP type will cure faster at room temperatures.

Excellent salt spray resistance of these solventless coatings along with the protection against attack by solvents and chemicals, points to their suitability in analogous environments.

Specialized Coatings

Cured fiberglass-polyester laminates are coated readily with the same formulations used for metal maintenance paints. Special solventless systems described by Hawley and Weber[16] for use on printed circuits have good adhesion to both fiberglass and copper, good mechanical strength and resistance to thermal shock and show good electrical resistance properties, particularly at high humidities over a broad temperature range.

Fiberglass-polyamide-epoxy systems are useful for repairing steel boats,

TABLE 10—Basic Formulations Suitable for Solventless Finishes

Material	Parts By Weight	
	Formula 1	Formula 2
Polamide 5	..	30
LVP	30	..
Triphenyl phosphite	3	3
Epoxy 2	70	70
Cardolite [1]	2	2
Silicone Fluid [2]	(3 ppm)	(3 ppm)

[1] Cardolite NC-513, Irvington Division, Minnesota Mining & Mfg. Co., Irvington, New Jersey.
[2] SF69, General Electric Co., Waserford, N.Y.

Polyamide-Epoxies

particularly where there is danger of undercutting of metal by corrosion.

Likewise, an effective method for repairing breaks in oil, gas and brine well casings involves application by means of an appropriate tool of a polyamide-epoxy fiberglass "patch."

Use of the polyamide system as a coating for baked phenolic surfaces has been well established.[17] One epoxy resin supplier has published several formulations for this application.[18]

Aluminum can be coated, particularly as indicated above, if the surface first is etched with hot chromic acid. Also, Alodine-treated surfaces give good results.

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18. Technical Bulletin, Release No. 16, Resins for Surface Coatings, Advance Technical Information, October, 1956, Bakelite Co., A Division of Union Carbide and Carbon Corp., New York, N. Y.

DISCUSSIONS

Question by Ignatius Metil, Buffalo, New York:

How does heat distortion temperature of an epoxy resin cured with a polyamide compare to the heat distortion temperature of the same resin cured with an amine, e.g., tetraethylene pentamine?

Reply by Don E. Floyd:

Typical heat distortion values for the polyamide-epoxy products, as compared with amine-cured epoxy resins, using a standard type of epoxy resin (Gen-Epoxy 190, Epon 828, Araldite 6010, or Dow Epoxy Resin 331) are:

Polyamide Product	Heat Distortion Temp., F
Versamid 115	140
Versamid 125	165
Versamid 140	230
Amine Curing Agents	
Diethylene triamine	240
Triethylene tetramine	240
Tetraethylene pentamine, data not available.	

Question by Irvin Baker, Baltimore:

What surface preparations were used prior to application of the Versamid epoxy coating on steel ship decks and on exposed angle irons?

Reply by David E. Terry:

Angle irons were wire brushed to remove loose scale, degreased and the coatings applied.

For the ship deck coating, the only preparation was steel wire brushing. The pigmented coating system was roller-coated on the deck, fiberglass blown onto the wet coating with a chipping gun and the topcoat roller-coated over the fiberglass mat.

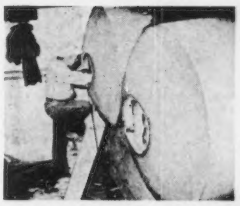
TABLE 11—Tests of Solventless Base Formulations in Corrosives

Corrosive	Formula 1		Formula 2	
	Condition	Days	Condition	Days
Vegetable oil	OK	240	OK	240
Water	OK	240	OK	240
NaOH 5 Percent	OK	240	OK	240
Trisodium phosphate 5 Percent	OK	240	OK	240
H_2SO_4 5 Percent	OK	240	OK	240
Lactic Acid 5 Percent	Slightly wrinkled (1)	240	Slightly wrinkled (1)	240
Acetic acid 5 Percent	Blistered (2)	8	Blistered (3)	8
Toluene	Wrinkled	1	Wrinkled	1
Gasoline	OK	240	OK	240

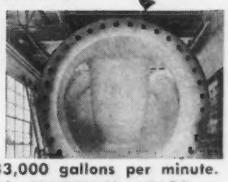
(1) No wrinkling 120 days.
(2) OK for three days.
(3) OK for one day.

Only

PLASITE*



* NO. 7155 COLD SET COATING



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"P-S" is the word these days, as engineers take this modern approach to low-cost, long-term corrosion protection. To learn more about P-S Constructions, write for "The Penton Buyer's Guide," a complete listing of suppliers of valves, pipe and fittings, pumps, meters, tank linings and coated parts made with Penton.

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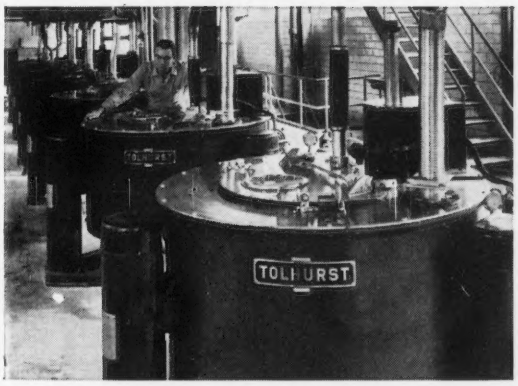
Engelhard Hanovia achieves excellent bonds between metal and Penton with its exclusive pipe-coating process. Thicknesses in the range of 10 to 60 mils can be easily obtained by this process.

PENTON SHEET WAS THE DOOR TO SOLVING GLIDDEN'S CORROSION PROBLEMS. In service more than 18 months at Glidden's Baltimore, Maryland, plant, this Penton-lined condenser door has been continually exposed to hot steam condensate with traces of sulfuric and other chemical additives. During operations, hot steam is drawn into the condenser from a large processing vessel containing 20% sulfuric, water and a mixture of titanium dioxide and iron sulfate at 230°F. The condensate is returned to the vessel, where a constant level and specific gravity are maintained. Rubber Millers, Inc., Baltimore, Md., manufactured the Penton-lined door for The Glidden Company.



FOR THE PROCESS INDUSTRIES... PENTON-COATED CENTRIFUGES. Pfaudlon 301 water suspension Penton coatings proved a "natural" for Tolhurst centrifuges which play so great a part in chemical processing. Practically every interior part of the centrifuge in contact with slurry, separated solid or liquid is coated with Penton to maximize resistance to corrosion. Penton coatings achieve the excellent bond strength needed for use in centrifuges where material constructions undergo tremendous stress. Pfaudlon 301 coatings are applied by Pfaudler Permutit, Inc., Rochester, N.Y., for Tolhurst Centrifugals Division, American Machine & Metals Inc., East Moline, Ill. Wherever processing conditions require corrosion resistance at elevated temperatures, Penton-Steel constructions are the logical means of achieving low equipment costs.

P-S YOU'LL SAVE MONEY

QP61-9

Polymers Department

HERCULES POWDER COMPANY
INCORPORATED

Hercules Tower, 910 Market Street, Wilmington 99, Delaware

Abstract

Describes serious pitting corrosion attack in autoclaves at plant where uranium ore is treated by leaching with sodium carbonate-bicarbonate solution. Serious pitting began after plant was on-stream for only six months. Attack was on mild steel vessels above liquid level.

Crash repair and corrosion protection program was begun while investigation was underway. Inconel strips were welded to froth line levels where pitting had occurred, but these patches also corroded. Coatings also were used in crash repair program: baked phenolics, epoxy-phenolic combinations, epoxies and vinyls. None of organic coatings tested was completely resistant to attack in vapor and liquid phase or pulp cones of vessels. Stainless steel belts then were welded to autoclave shells where pitting had occurred.

Later, more rapid corrosion rates were encountered, no longer confined to froth line of the autoclaves. Corrosion spread to classifier shafts in the grinding area, pitting of filter drums and even affected clarifier frames in contact with clear solution. Pachuca tanks also were subject to pitting. Study showed major cause of this increased corrosion was a change in alkalinity of mill solutions. Investigators recommended a minimum mill solution pH of 10.0. Describes satisfactory protection service of epoxy coatings in Pachuca tanks after three years' operation.

3.22, 8.10.2, 5.4.5

Figure 1—Autoclaves used at Beaverlodge Plant to leach uranium ore. These vessels had serious pitting corrosion above the liquid level after less than six months' service after the plant was put into operation.

SERIOUS PITTING corrosion on process tanks after only a few months' operation at the Beaverlodge Processing Plant forced Eldorado Mining and Refining Limited, Ottawa, to begin a crash program of corrosion control while an investigation was made of the specific causes of the problem.

To explain the serious corrosion problem, this article outlines the process of uranium ore treatment at the plant, describes the equipment which was corroded badly and explains the control measures taken by the plant personnel to correct the problem.

Uranium Ore Treatment

The Beaverlodge Processing Plant treats uranium ore by leaching with sodium carbonate-bicarbonate solution followed by precipitation of uranium with caustic soda as a sodium di-uranate. The barren solution is carbonated to convert excess caustic soda to carbonate and bi-carbonate; this solution is returned for leaching of fresh ore. Considerable build-up of reaction products occurs because the only solution lost from the plant is that which remains in tailings filter cake after washing with water. Solution lost in the filter cake is only six to eight percent of daily tonnage of leach solution.

Leaching is done in two types of equipment: autoclaves and Pachuca tanks.* High temperatures and thorough aeration of the pulp are requisite conditions for satisfactory leaching rates.

The autoclaves, shown in Figure 1, are horizontal cylinders of mild steel with dished ends. Eight feet in diameter and 25 feet long, the autoclaves are arranged in two parallel rows of nine tanks each. Material of construction is plain carbon steel (AISI 1020). These tanks are operated with about 80 percent liquid volume. Air fed into the tanks at 550 standard cubic feet per minute to each row is dispersed in the pulp by aerators. Two agitators are used to mix and suspend the pulp.

The Pachuca tanks are 18 feet in diameter and 50 feet high with 60-degree cone bottoms. These tanks also are made of mild steel. Air is sparged into the tanks through pipes at 125 standard cubic feet per minute per tank. The tanks are arranged in four parallel rows of six tanks each.

Ore treated at the Beaverlodge Plant contains feldspar rock, quartz, calcite, chlorite and pyrite. The ore is ground to 75 to 80 percent minus 200 mesh, and the slurry densities during leaching are 48 to 54 percent solids. Pulp temperature is 170 to 175 F in the Pachuca tanks and 235 F in the autoclaves.

The mill solution contains carbonate, bi-carbonate and sulfate of sodium. Other salts present during leaching include sodium uranyl tri-carbonate, sodium chloride and small amounts of vanadium.

Serious Pitting Problem Occurs

After just a few months' operation in 1953, inspection of the autoclaves showed serious pitting corrosion of the mild steel above the liquid level. This corrosion was associated with adhering deposits of caked slime near the froth level. This carbuncular deposit is shown in Figure 2. Figure 3 is of the same tank area after gritblasting.

*A cylindrical, cone bottomed tank with a central cylinder for introduction of compressed air, used in agitation and leaching of pulp. These tanks were originally devised in New Zealand but first practical introduction was in Pachuca, Mexico.

crash repair program to control SERIOUS PITTING CORROSION on process tanks*

G. F. Colborne, A. R. Allen and A. Thunaes

Eldorado Mining and Refining Limited
Ottawa, Ontario
Canada

Battelle Memorial Institute was engaged to study the corrosion problem, but the seriousness of the pitting rate made it necessary that protective coatings and linings be installed immediately in an attempt to control the corrosion.

Battelle Institute concluded that froth line corrosion was caused by froth, enriched with sulfides, deposited through pulp splashing. The protective alkaline solution was removed by condensate drained from the wall and by reaction with sulfides. Subsequently, the sulfides oxidized to sulfurous and sulfuric acid which promoted corrosion. Differential aeration corrosion cells were formed at the surface of the metal under the mud splashes. Under such deposits, local attack was stimulated if there were other depolarizers in addition to oxygen to promote

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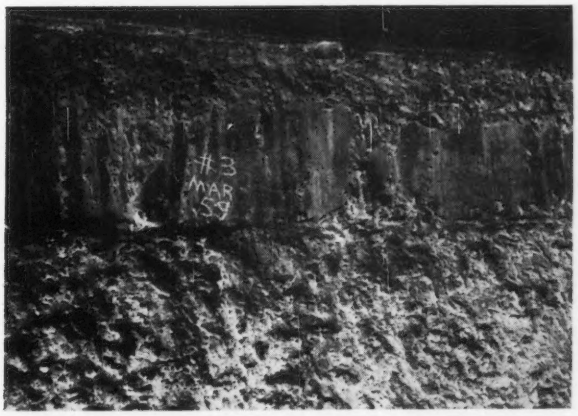


Figure 2—Typical carbuncular deposit on autoclave wall.

★ Revision of a paper titled "Corrosion Research and Control in Eldorado's Beaverlodge Mill" presented at the 17th Annual Conference, National Association of Corrosion Engineers, Buffalo, N. Y., March 13-17, 1961.

serious pitting corrosion—

(Continued From Page 21)

reaction of local cathodes. Ferric iron and graphite were mentioned as possible depolarizers.

This froth line corrosion was the only type of serious corrosion in the original mill which was expanded to a capacity of 700 tons per day by addition of four Pachuca tanks in 1954 and to 2000 tons per day in 1957.

Repair and Protection Program

Plant equipment repair and protection program involved only the autoclaves or pressure leaching vessels for the first two years, 1953-54. At that time the corrosion of Pachucas, grinding circuit classifiers, filters and pregnant solution clarifiers was becoming increasingly serious. Protective measures on the latter equipment and the increased alkalinity of mill solutions to reduce corrosion resulted in continuous operation of these units with minimum cost and downtime.

The autoclaves, however, presented a more serious problem because they were the first vessels attacked and because little was known at that time about the corrosion mechanism involved or satisfactory protective measures.

With the discovery of serious pitting at the froth line of the first four autoclaves in each bank after less than six months' operation, a crash repair program was begun.

After consultation with representatives of Battelle, International Nickel Company and Mines Branch of the Canadian Department of Mines and Technical Surveys, an Inconel strip (12 inches wide and ⅛-inch thick) was welded with Inco "A" rod to the autoclave shell at the froth line, as shown in Figure 4. Six vessels were repaired in this manner. Within one month's operation, corrosion of the Inconel was very evident. Pitting occurred under the ore pulp splashes or carbuncles and penetrated the Inconel plate to a depth of about 40 percent of its thickness.

Coatings in Crash Repair Program

A secondary emergency repair program was begun, using a baked phenolic formulation as a protective coating. Each of the previously repaired six autoclaves was gritblasted and given four coats of primer and two coats of clear resin over the complete upper half of the vessel. Each coat was partially cured; final cure was eight hours at 300 F. After one month's service, the coating below the liquid level had disappeared completely but was still bonded and providing protection in the vapor phase area.

A series of panel evaluation studies of over 50 possible repair materials was begun by Battelle in one of the production autoclaves. To check the theory that sulfides in the froth at the liquid surface were the major factor in the rapid corrosion rate, a small pilot scale operation was set up involving removal of sulfides by froth flotation and leaching of subsequent flotation tailing. A second pilot-scale autoclave operating on fresh feed was used as a control.

The first six autoclaves which had been partially lined with Inconel and coated with phenolic material were left on stream. Repairs were made to other vessels farther down the line. Procedure adopted was to weld a 24-inch strip of ¼-inch mild steel plate around the autoclave at the froth line. Eight more vessels were repaired in this manner; two of these were protected further by application of organic coatings in a comparison test. On one, the phenolic formulation was used; on the other an epoxy-phenolic formulation. After 47 days on-stream, both coatings were failing. As before, the phenolic coating was intact in the vapor phase but destroyed in the liquid area. The epoxy-phenolic also was resistant in the vapor phase but badly blistered in the liquid area.

Results of the panel evaluation studies showed that stainless alloys, particularly Type 316 and Type 347, were unaffected by six months' exposure in a production vessel. The mild steel to stainless steel welds were resistant, and there was no evidence of galvanic attack.

None of the organic coatings tested was completely resistant to attack in the vapor and liquid or pulp cones. In general, the epoxy and vinyl types wrinkled or blistered and peeled; most of the bitumastic and rubber coatings became hard, cracked and then flaked off.

On basis of this preliminary information from panel evaluations, another system was selected for testing on a production autoclave. A modified phenolic resin polymerized by a catalyst at room temperatures was applied to four autoclaves as one primer, one body and two seal coats, giving a total thickness of about 40 mils. Curing was 36 hours at 90 F.

This modified phenolic system gave fair protection for two to six months. In most cases the topcoat flaked off, but the primer gave protection for longer periods.

Two more autoclaves were coated with a modified phenolic and neoprene system in hopes that the neoprene topcoat would enhance the reasonably satisfactory performance of the phenolic primer and body. After two months' service, the neoprene was flaking badly over about 50 percent of the area.

At this stage of the investigation and repair program (in the fall of 1955) further testing of organic coatings was abandoned. Installation of stainless steel belts around the vessels at the froth line was continued. Only the first three or four autoclaves in each bank of nine would require this degree of protection; earlier inspections showed that severity of attack was reduced in vessels towards the end of the line.

The first autoclave was repaired with a 36-inch wide belt of 3/16-inch thick stainless steel clad plate, having 30 percent cladding of Type 316L stainless. All welding to the shell was done with Type 316 ELC electrodes. Width of stainless belt was reduced to 30 inches in succeeding autoclaves.

During installation of these stainless belts in the third autoclave downstream, severe pitting was found in the bottom directly below the center aeration impeller. Some pits had penetrated ½-inch in the ⅝-inch plate. Immediate inspection of all autoclaves revealed pitting in most of those in the middle and end of the line with the severity increasing toward the end. One of these autoclaves was repaired by welding a ⅜-inch mild steel plate under the center impeller, but within three months this clean plate was pitted to a depth of about ⅛-inch.

Corrosion Problem Becomes More Serious

Corrosion was no longer confined to the froth line of the first three autoclaves in each row, as previously experienced, but was most serious in downstream autoclaves below the pulp surface. Corrosion spread to many sections of the plant, caused failure of classifier shafts in the grinding area, pitting of filter drums and even affected clarifier frames in contact with clear solution. The Pachuca tanks, not previously at-

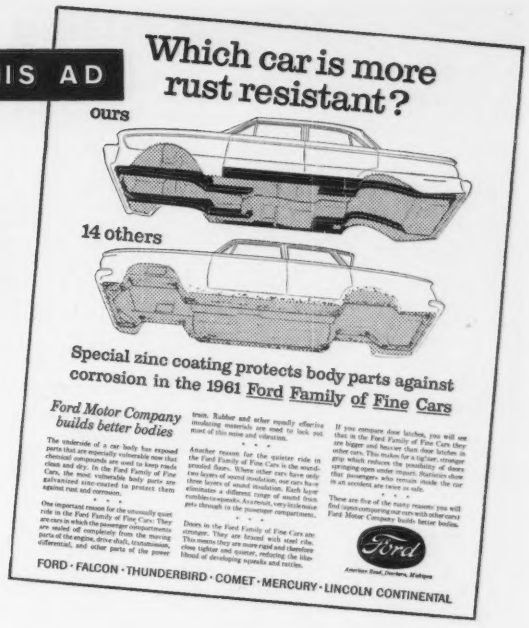
(Continued on Page 24)

Figure 3—Same area of autoclave wall as shown in Figure 2 after grit-blasting.

THE RESEARCH STORY BEHIND THIS AD

ZINC

—Chosen By Process Of Elimination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A — **ORGANIC COATINGS**
1. Primers (Solvent Base and Water Emulsion Base)
2. Metallic Rich Paints
3. Plastic Coatings
4. Self Foaming Materials
5. Greases, Waxes, etc.

B — **STEEL ALLOYS**
1. Stainless Steel
2. Special Alloys

C — **MORE UNUSUAL METHODS**
(Such as vapor phase inhibitors, irradiated steels, forced ventilation, etc.)

D — **METALLIC COATINGS**
1. Aluminum Clad
2. Terne Plate
3. Tin Plate
4. Cadmium Plate
5. Zinc Coated
   A. Electrogalvanized
   B. Hot Dipped

Reprints of an extensive report on the Ford research program which culminated in their choice of Zinc are available on request from St. Joe.

The search for the best defense against corrosion was a major project with Ford Motor Company. A long and painstaking series of laboratory, road and production tests were scientifically conducted and evaluated before Ford named the winner—*galvanized steel.*

FORD EVALUATED THEM FOR

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2. Formability
3. Weldability
4. Painting
5. Method Employed (special requirements in applying, inspection, etc.)
6. Stock (and scrap) handling problems, if any
7. Cost

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—AND THE ZINC FOR THE JOB

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ZN-192

serious pitting corrosion—

(Continued From Page 22)

tacked, now were subject to pitting corrosion.

An intensive study was begun to determine the cause of this widespread corrosion. Records were examined to determine changes in operating conditions that had been made in 1956. The only major changes were in the alkalinity of mill solutions; minor changes were noted in grinding, pulp density and tonnage per unit.

The Mines Branch of the Canadian Department of Mines and Technical Surveys and Battelle Memorial Institute were engaged to study this corrosion problem. Both agencies reported on the effect of solution composition on the corrosion rate.[1,2] Reduction of mill solution pH seemed to be principal cause of the greatly increased corrosion rates during 1956-57. The general conclusions were as follows:

(1) Oxygen concentration cells were responsible for pitting corrosion.

(2) Such cells are particularly active when a part of the metal surface is covered with mud cakes or corrosion products while a large area of adjoining base metal is in contact with aerated solution.

(3) Sulfate and chloride ions increase the corrosion rate, particularly if solution pH is less than 9.5.

(4) Pitting corrosion is not observed (or is insignificant) when solution pH is 9.7 or higher.

These conclusions on protective alkalinity agreed with observations that mild steel tanks containing pregnant or barren solution of high pH had never shown signs of pitting corrosion. Also, American carbonate leaching plants which maintained a high pH during leaching, did not have corrosion trouble. Mud deposits are not required to initiate and promote pitting corrosion. Test panels in contact with clear carbonate solutions at room temperatures show spectacular corrosion rates when solution pH is 9.5 or less.[1] Large pustules are formed and heavy pitting corrosion takes place under these pustules.

The function of protective alkalinity as a means of stopping corrosion on mild steel is not understood completely, but it has been found that corrosion products are less soluble in solutions of pH above 9.5. The coating formed by these products is less permeable to passage of oxygen and therefore more protective. Alkaline solutions above this value tend to passivate the iron. Corrosion rates decrease in proportion to hydroxide concentration and amount of salts in solution. Salts in solution decrease the beneficial effect of alkalies.[3]

Higher pH Lowers Corrosion Rate

As soon as the detrimental effect of low alkalinity was demonstrated in the laboratory studies, the mill staff increased the mill solution pH to a minimum of 9.5. Experience over the last three years has shown that this higher alkalinity has eliminated serious corrosion by clear solutions and has reduced the corrosion rate in leaching vessels containing ore pulps.

Figure 5—Craze-cracking mosaic pattern that occurred on a modified phenolic coating system applied to Pachuca tank. This effect probably was partially caused by vessel movement.

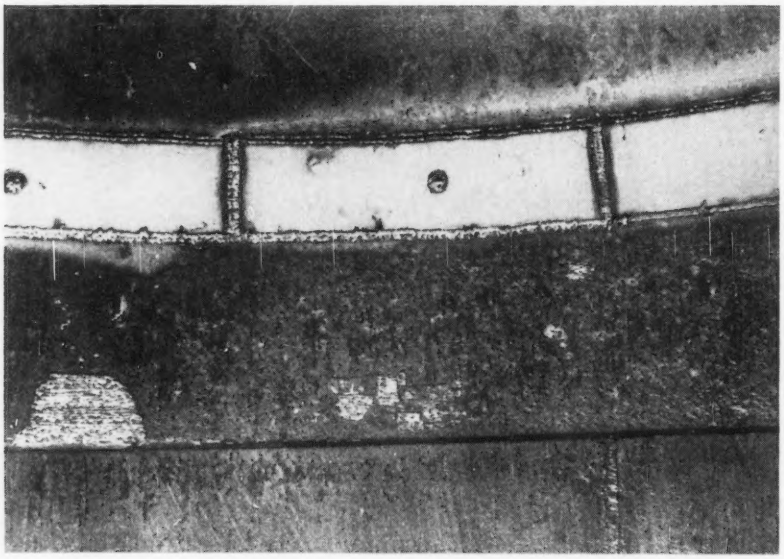

Figure 4—Inconel strip was welded to autoclave shell at the froth line where pitting corrosion was most severe, but after one month's service, this welded plate had corroded.

For economic reasons, the Beaverlodge Plant could not be operated with a solution of pH of 10.0, as recommended. A minimum pH of 9.5 set as a compromise has given good protection at reasonable cost, but changes in feed characteristics may occasionally cause a temporary drop in pH below the desired level.

Repairs to autoclaves continued on a regular schedule with each vessel in turn having a ⅜-inch mild steel plate welded over the complete lower half of the vessel and three-foot square patch of Type 316 stainless welded in the center bottom area. In a few vessels, more serious pitting was found on other areas of the shell; in each case of pitting, a 1/16-inch thick Type 316 plate was used to protect the area.

At present, autoclaves are inspected every nine months. Any necessary repairs are done with 1/16-inch thick stainless sheet. During the past year or so, pit measurements and photographic evidence indicate that little corrosion is occurring.

Protection of Other Equipment

On other mill equipment such as Pachuca tanks, classifier shafts, filters and clarifiers, organic coatings have been giving good protection. The four original Pachucas installed in late 1954 were repaired by welding mild steel plates in the area of the cone-vertical wall intersection where corrosion was found to be concentrated. Three of these vessels and the 20 new ones installed in 1957 were coated in this area with the modified phenolic system. On the other vessel, epoxy was applied as a test.

The modified phenolic system is resistant to attack in the Pachucas but forms a brittle coating which tends to crack in a mosaic pattern of craze-cracking, as seen in Figure 5. Vessel movement probably is partially responsible for this effect.

The epoxy is giving excellent protection after three years' service in the Pachucas. A program is under way to replace all phenolic coatings with epoxy. Epoxy also is being applied to many tanks, filters and clarifiers. These epoxies have been satisfactory for continuous contact with carbonate solutions and slurries at temperatures to 180 F.

Acknowledgment

The authors are indebted to representatives of Battelle Memorial Institute, particularly W. K. Boyd, F. W. Fink, E. L. White and C. L. Peterson, from whose unpublished progress reports much of this data was obtained.

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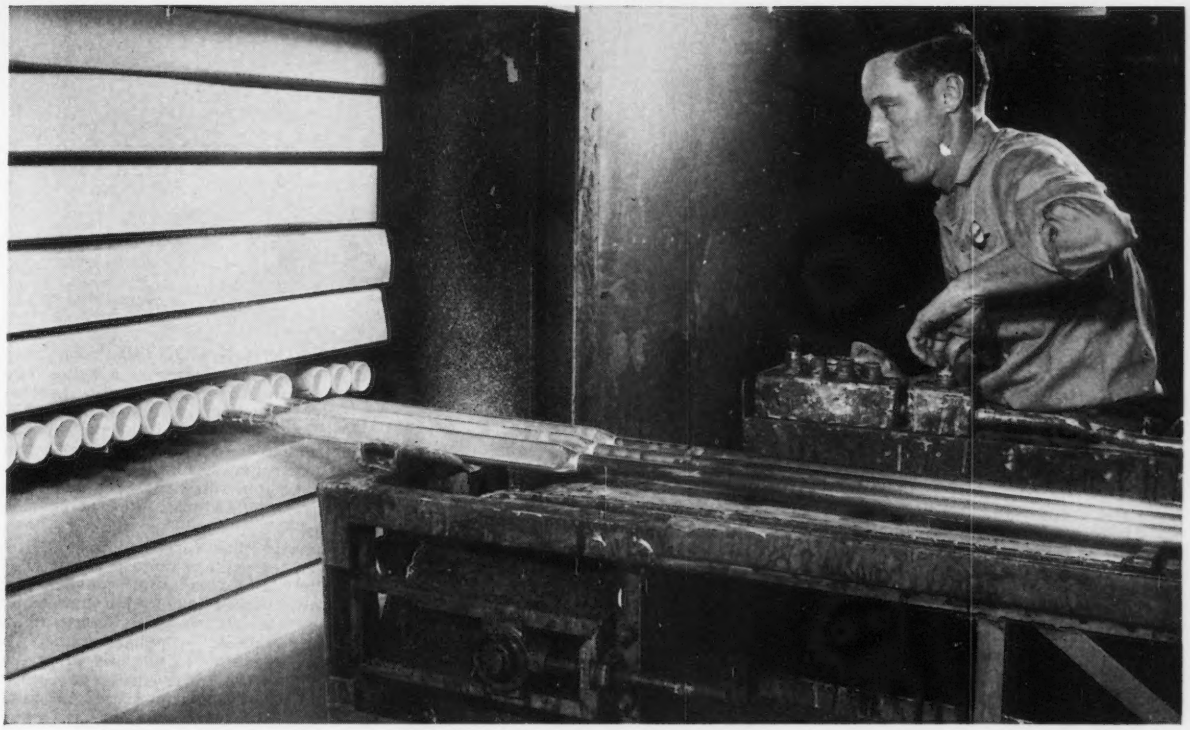

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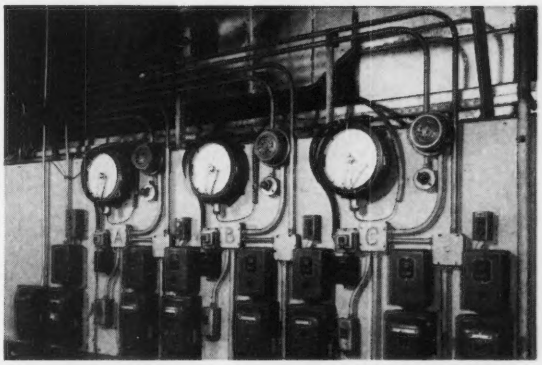

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BETTER COATINGS THROUGH RESEARCH

PLASTIC APPLICATORS, INC.

Charles W. Holt
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cell apparatus checks resistivity of nitrogen tetroxide propellant to control CORROSION in MISSILES*

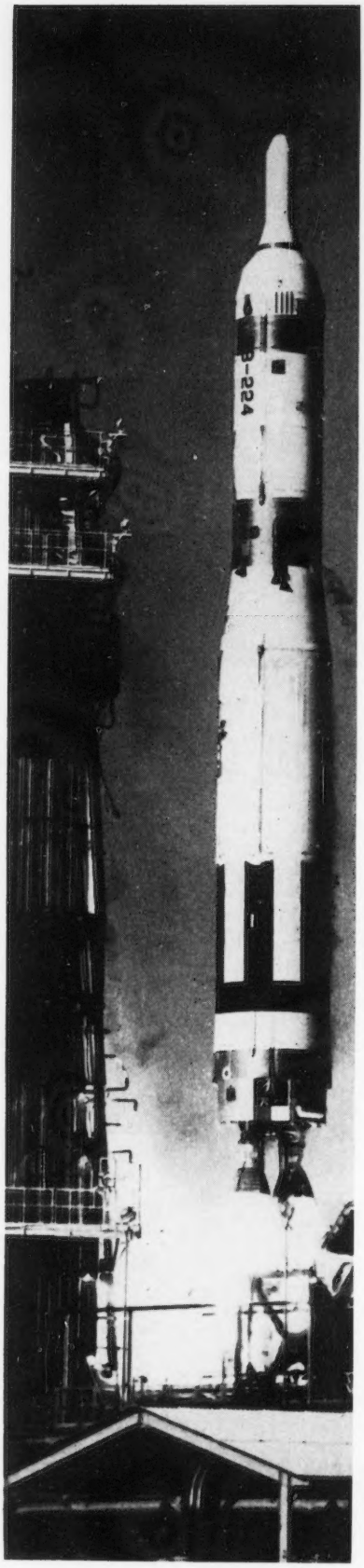


Figure 1—Titan ICBM being launched at Cape Canaveral. Titan is produced by the Martin Company's Denver Division.

CORROSION CONTROL on intercontinental ballistics missiles with nitrogen tetroxide propulsion systems is achieved principally by regulation of water content. Most of the materials of construction used for storage and transfer of nitrogen tetroxide propellant resist corrosion sufficiently if the water content is below about 0.2 percent by weight.

Materials that resist corrosion from nitrogen tetroxide include stainless steel, aluminum alloys and some plastics such as tetrafluoroethylene. Aluminum alloy 2014-T6 has been used for construction of some missile propellant tanks for nitrogen tetroxide, but this alloy requires that the most stringent control be maintained on water contamination of the propellant to minimize corrosion by the missile propellant. These aluminum alloy tanks are designed to handle the missile propellant for long periods until the missile is fired. Water content above the specified 0.2 percent by weight can cause corrosion and subsequent destruction of the missile.

Nitrogen tetroxide propellant is termed "storable" as opposed to the standard missile oxidizer-liquid oxygen. Principal difference between the two propulsion systems is that a missile can be loaded with nitrogen tetroxide propellant and maintained in a ready condition for quick firing. With missiles using liquid oxygen, loading can be done only just before launching because the —297 F boiling point of liquid oxygen produces uncontrollable material loss.

A new approach to determining water content in missile propellants was investigated because current methods used in industry to determine water content require periodic sampling and extensive analysis time in a chemical laboratory. Both of these requirements, obviously, would be unsuitable for checking water content in missile propellants, especially when the missile is stored in an underground silo as part of the nation's defense system.

Studies of electrical resistance properties of nitrogen tetroxide showed that a definite relationship exists between water content and specific resistance of the propellant. Further investigation resulted in the design and construction of a cell apparatus from which a curve can be plotted showing water content in relation to specific resistance.

Resistance is a general property of all liquids. Specific resistance or resistivity has the dimension of ohm-centimeter

★ Submitted for publication March 23, 1961.

Abstract

Brief discussion of cell apparatus used to make continuous analysis of water content in nitrogen tetroxide propellant systems as a method of corrosion control in intercontinental ballistics missiles. Aluminum alloys used for missile propellant tanks handling nitrogen tetroxide suffer severe corrosion when water content in system rises above 0.2 percent by weight. Studies showed relationship between water content and specific resistance of missile propellant. Discusses formula for determining specific resistance of a liquid. Shows equipment used for this method of water content analysis and outlines its advantages over laboratory analysis methods.

4.3.7, 6.4.2, 7.1

and is measured at a selected temperature. Specific resistance is defined by the expression

$$r = \frac{RA}{L}$$

where R is the resistance in ohms of the liquid, A is the area in square centimeters of the facing of the electrodes, L is the distance in centimeters separating the electrodes.

As resistance is increased, sensitivity of the measuring equipment must also be increased. This can be done in one of two ways: first by increasing the sensitivity of the Wheatstone bridge and second, by decreasing the cell constant K of the measuring cell. Resistance indicated by the meter divided by the cell constant is equal to the true specific resistance. As K decreases, a higher specific resistance can be measured with the same bridge.

The first method is not feasible because, as the measurable resistance increases beyond 10^9 megohms, interference with subseqeunt loss in accuracy of the bridge is encountered by stray electrical fields, unshielded wire, resistance of leads, etc. Expensive equipment using Faraday cages are available but are not adaptable for measurement of nitrogen tetroxide because of its corrosive properties.

The second method (decreasing cell constant) is feasible. For example, if the distance between the electrodes is made constant at one millimeter, a change in the surface area of the electrodes will cause an inversely proportional change in cell constant. Cell constant values of any magnitude can be obtained in this manner with cell size being the only limiting factor.

Specific resistance measurements of nitrogen tetroxide require equipment that can measure in the magnitude of 10^7 megohm-cm. A Wheatstone bridge, galvanometer, Ayrton shunt and a specific resistance cell were designed for use with nitrogen tetroxide. The bridge was operated by direct current and could measure resistances to 10^9 megohms. The galvanometer, operated through an Ayrton shunt to regulate sensitivity of the input current, was used as a visual means of determining the balance of the bridge.

A cell was constructed of three concentric shells equally spaced one millimeter apart with sufficient surface area to give a cell constant of 10^{-4}. This cell constant provided a capability of measuring in the 10^7 megohm-cm range.

This cell apparatus technique for determining water content in nitrogen tetroxide can be used in the laboratory as an analytical procedure and on missiles for continuous monitoring of water content in the propellant storage and transfer systems.

Analytical procedures currently used by industry apply gravimetric or freezing point techniques which require two to six hours to analyze one sample. Accuracy of these methods is affected by the same factor which limits use of the cell—contamination in the nitrogen tetroxide other than water such as metal ions, corrosion products, and dissolved organics. These contaminants can affect specific resistance readings, freezing point determinations and gravimetric assays. Therefore, specific resistance techniques are not recommended except for analyzing commercially pure nitrogen tetroxide.

Principal advantage of using the specific resistance method in the laboratory is that a sample can be analyzed in five to ten minutes (as compared with two to six hours by normal techniques) and is as accurate as normal analytical methods.

When used to check water content on missile propellant storage and transfer systems, the specific resistance method provides a quick and continuous monitoring of water content. Data can be recorded on a strip recorder or periodic readings can be made without obtaining a sample. Corrective action can be taken in the event of accidental contamination before extensive corrosion damage to the missile occurs.

To monitor a storage or transfer system by standard methods, samples must be drawn from the system and sent to a laboratory for analysis. Possible errors are introduced through sampling and handling. This can become quite a problem if the propellant system is stored underground. Also, knowledge of water content of the system is known only at the sampling intervals. No continuous monitoring could be provided by this method.

In the missile industry where high copper aluminum alloys are used as materials of construction in some of the missile propellant storage and handling systems and where frequent transfer and handling of a single batch of propellant are common, water content must be controlled as closely as possible. Only a continuous monitoring system such as the cell apparatus for determining specific resistance of the propellant can satisfy the need of the missile industry for this specific corrosion control problem.

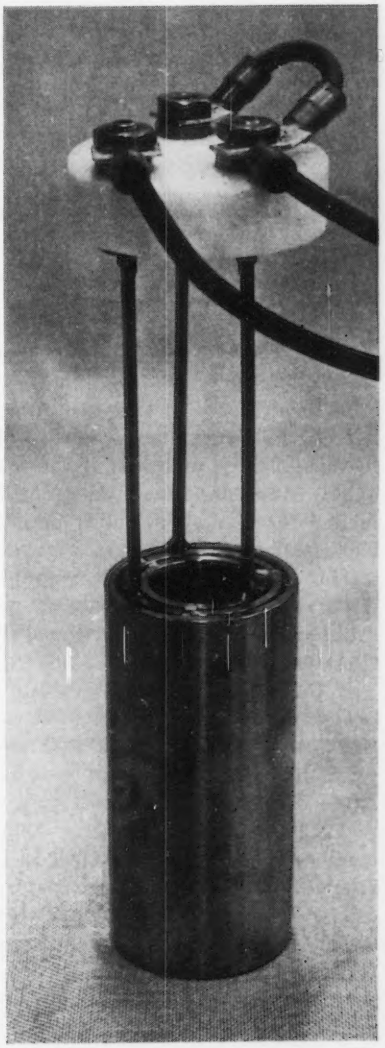


Figure 2—Cell used to determine water content in nitrogen tetroxide missile propellant. Resistivity can be determined in laboratory as analytical procedure or on missiles for continuous monitoring of water content in propellant storage and transfer systems to prevent corrosion. Cell has three concentric shells spaced one millimeter apart.

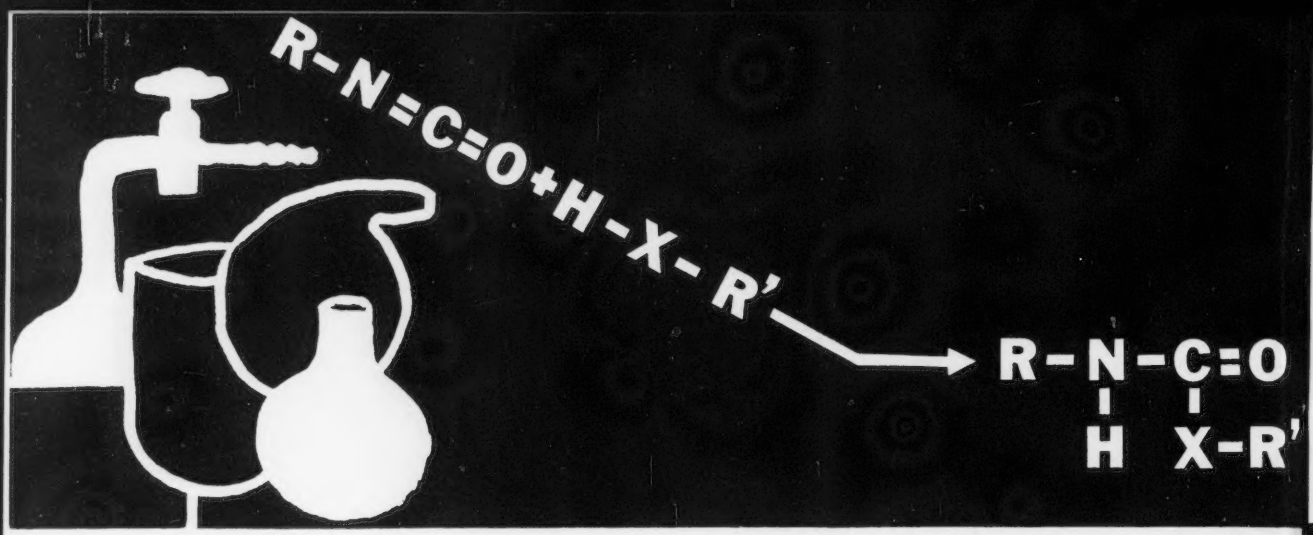


Figure 1—Basic urethane reaction. Product of reaction is a urethane if X is oxygen.

J. W. Cushing, John F. Montle

and

J. D. Byrd

Carboline Company
St. Louis, Missouri

Abstract

Describes the basic differences in major types of urethane surface coatings commercially available. Illustrates considerable variation in chemical resistance of commercial formulas in two-package or catalyzed urethane group. Describes superior resistance of catalyzed urethanes to softening by high-solvency organics as compared to commercial epoxy-amine coatings. Illustrates urethane retention of flexibility and impact resistance after weathering exposures as compared to commercial catalyzed epoxies. Describes briefly several types of urethane coatings that are being developed. 5.4.5

ISOCYANATE COATINGS is a better name for the group of coating materials that have been known variously as urethane, polyurethane, isocyanate or polyisocyanate type coatings. The common feature of these materials is the reaction of an isocyanate with an active hydrogen, especially one attached to an oxygen, sulfur or nitrogen atom. This basic reaction is shown in Figure 1.

The isocyanate most commonly used is tolylene diisocyanate which is reactive at both isocyanate groups. The resultant reaction forms a three dimensional structure in the coating film.

If the active hydrogen contributor is a hydroxyl group (if "X" in Figure 1 is oxygen) as in the reaction of an isocyanate with polyglycols, certain polyesters or oils, such as castor oil, the chemical name of the initial product is a urethane. It then reacts further with more isocyanate to form a more complex chemical structure. If the isocyanate is reacted with an amine, a urea type product is formed.

The isocyanate group will react directly with water, forming carbon dioxide gas and one of the final products, as illustrated in Figure 2.

In the use of a two-package urethane coating, if considerable moisture is in the materials or on the surface, or if moisture condenses from humid air onto the film while curing is taking place, it will "use up" some of the isocyanate portion. This changes the final chemical structure of the film (generally reducing both chemical resistance and flexibility), and the carbon dioxide formed may cause bubbles and pinholes in the film. Thus, in urethane systems with reactive isocyanate present, moisture contamination and wet atmospheres should be avoided.

Urethane coating types can be divided between air-dry and baking systems.

Baking Urethane Coatings

The diisocyanate can be converted to a non-reactive product by reacting it with phenol. This isocyanate-phenol product is mixed with a polyol and shipped as a single-package paint. After application, the film is baked at 300 F or higher. The bake drives off the phenol, and the isocyanate groups quickly crosslink with the polyol to form a hard, tough coating. This type coating can be formulated to retain adhesion and toughness to 300 F. Physical properties are better at 250 to 300 F than typical baked phenolics and baked epoxy-phenolics.

Laboratory Tests Show

Resistance Properties of Urethane Coatings*

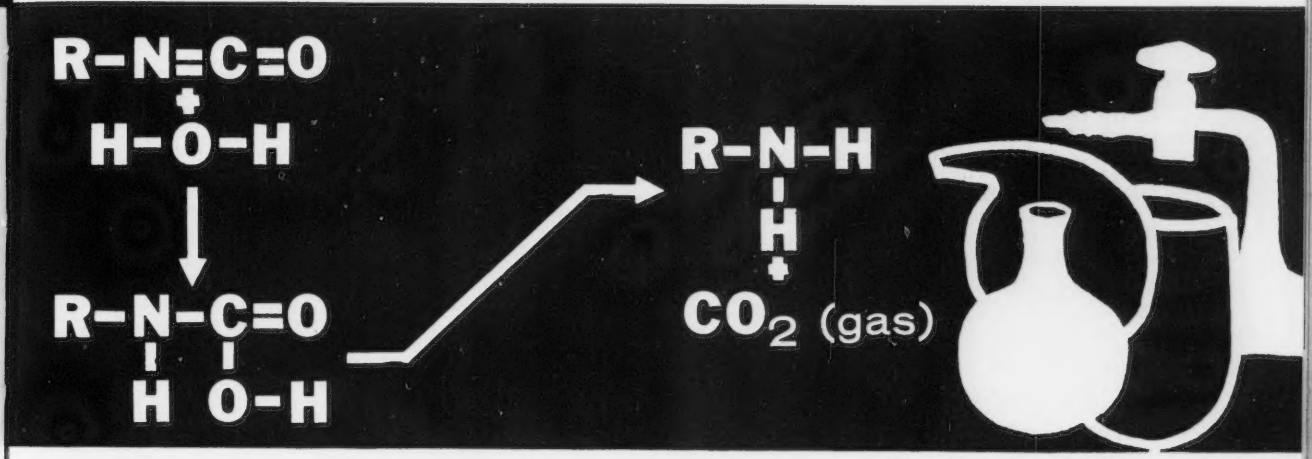


Figure 2—Reaction of isocyanate with water.

In organic solvent resistance, the baked urethane compares favorably with baked epoxy-phenolics but is generally inferior in acid and alkali resistance.

Electrical properties of the baked urethane film are excellent. Major use of this type is as a wire coating.

Air-Drying Urethane Coatings

The air-drying group can be subdivided into single component (one-package) and multiple component (chiefly two-package) systems: One-package types are urethane-oil paints and moisture reactive urethanes. No free isocyanate is present in urethane-oil paints, a relative of alkyd-oils, phenolic-oils and epoxy-esters. The isocyanate has been prereacted with a drying oil, such as linseed oil, in much the same way phenolic resins are cooked with tung oil to make the well-known phenolic-tung oil varnishes and enamels.

The high price of the isocyanate makes the urethane oil coating cost 30 to 50 percent more than conventional alkyd enamels, so the urethane must offer something unique. This coating's main feature is its ability to dry fast, a useful property to production-line users and on jobs where painting interferes with normal work. Fast dry alkyds and phenolic-oils generally require four to eight hours to dry dust-free; most urethane-oils dry within two hours.

Chemical resistance of urethane-oils is only moderate because of the large drying oil content. The coating behaves in acid or alkali fume exposures much like an epoxy-ester. Chalking rate is similar, too. The choice between a urethane-oil and an epoxy-ester should usually be made on the basis of differences in the individual formulas rather than because of generic type. Where fast drying is important, the advantage is with urethane-oil.

Unpigmented urethane-oils are useful as wood floor finishes and as spar varnishes. The clear film is chalk-resistant, dries fast and is one of the toughest oil-modified varnish types available.

Moisture Reactive Urethanes

In moisture reactive urethanes most of the isocyanate is prereacted by the manufacturer with a polyol. The coating has excess isocyanate which reacts with moisture from the air to harden the film. These coatings have limited use in field appli-

(Continued on Page 30)

★Revision of a paper titled "Comparative Properties of the Major Types of Urethene Coatings" presented at the 17th Annual Conference, National Association of Corrosion Engineers, March 13-17, 1961, Buffalo, N. Y.

urethane coatings—

(Continued From Page 29)

cation conditions because of varying humidity conditions. Each coat must be thin, 1½ mils or less, in order that film crosslinking will be complete and uniform. Major use is in shop applications as a one-coat finish over plastic and rubber surfaces.

Two-package urethane coatings, shipped as two separate components, are mixed in the field just before use in the same way that catalyzed epoxies are handled. Pot life after mixing varies from 1 to 48 hours, depending on the individual formula. Members of this group in current use are urethane-coal tar and isocyanate adduct-polyol coatings.

Urethane-Coal Tar Coatings

In urethane-coal tar coatings the reactive hydrogen groups in certain coal tar pitches are crosslinked with isocyanate (generally used in the form of a prereacted adduct of diisocyanate and a polyol). Crosslinking the tar in this manner gives a striking improvement in solvent resistance of the tar so that the product is not deteriorated by direct contact with aliphatic fuels. If the formula has a high isocyanate content, the acid and alkali resistance are of the same order as amine-cured epoxy-tar coatings, but the cost is a little higher than epoxy-tars.

This type coating is useful in water immersion conditions, on below-grade surfaces and in heavy spillage areas where the thick film and good chemical resistance of coal tar pitches suggest their use, but where a coating with greater toughness or solvent resistance than straight coal tar is desired. Obviously there is overlapping of urethane-tar coatings and epoxy-coal tars in areas of use. Individual formulas among these types should be investigated or compared to make a selection.

Isocyanate Adduct-Polyol Coatings

The most common type of two-package urethane coating is isocyanate adduct-polyol. One component is a prereacted adduct of an isocyanate with a polyol, as a polyglycol. The most important reason for supplying the isocyanate portion as a prereacted polymer is to minimize the content of volatile tolylene diisocyanate so that coating toxicity is greatly reduced. The second component is a polyester, polyether or oil, such as castor oil. After mixing, the polyol crosslinks with unreacted isocyanate to form an insoluble, tough, thermosetting film.

The newly applied coating resembles an epoxy-amine or epoxy-polyamide: it is hard, usually flexible and glossy. Original gloss of most two-package urethanes is probably the highest of any of the chemical-resistant synthetic coatings. In outdoor exposures, pigmented films chalk rapidly, so that after a year's exposure their appearance is the same as epoxy-amines. Clear urethanes are unusually chalk-resistant, and in some cases a clear two-package urethane is applied over the pigmented coating.

Yellowing of the film during outdoor exposures, a characteristic of two-package urethanes, is noticeable in white and pastel shades. The clear coating turns yellow also, but this may not be noticeable over deep colors. A few commercial urethanes show only minor yellowing, generally because they contain less isocyanate. The manufacturer's advice should be followed on outdoor colors.

Most of the commercial two-package urethanes give low film build per coat. Careful spraying in the field will produce 2 mils dry film, but brushing yields only 1 to 1½ mils. Manufacturers are working on high-build formulations to give 3 to 6 mils per coat. The first of these are available.

Properties of Two-Package Urethanes

Properties of the two-package urethane coatings to be discussed in this article

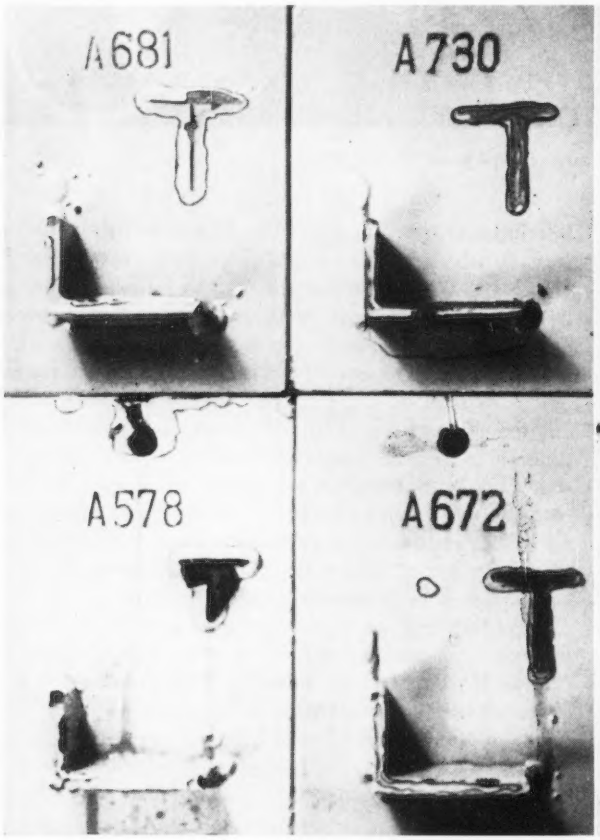


Figure 3—Test panels exposed in hydrochloric acid vapors at 75 F to comparable failure. Panel A681 was 6 mil epoxy-amine exposed 18 weeks, Panel A578 was 6 mil vinyl exposed 13 weeks, Panel A730 was 5 mil urethane exposed 9 weeks, and Panel A672 was 5.5 mil urethane exposed 9 weeks.

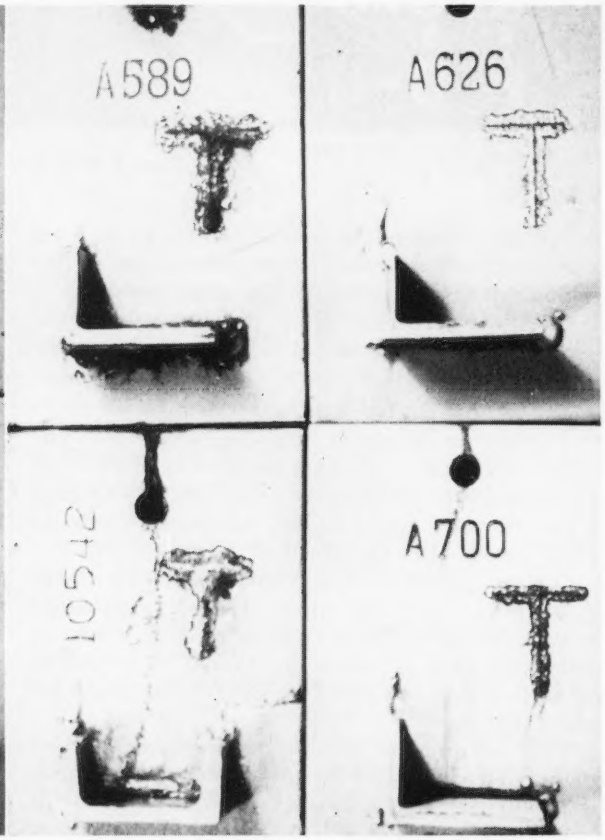


Figure 4—Test panels exposed in salt fog cabinet (synthetic sea water at 110 F) to initiation of edge failure. Panel A589 was 6 mil vinyl exposed 18 weeks, Panel 10542 was 6.5 mil epoxy-amine exposed 21 weeks, Panel A626 was 5 mil urethane exposed 9 weeks, and Panel A700 was 5 mil urethane exposed 9 weeks.

include chemical resistance, film flexibility retention, bonding and compatibility with existing paints. Data from laboratory panel tests are given to illustrate these properties.

Chemical Resistance

General chemical resistance of these coatings is comparable to the best resin types available. Useful in acid, salt, water and organic solvent exposures, the urethanes are similar to the resistance characteristics of the amine cured epoxies. In strong alkali conditions, the performance of urethane systems varies widely with the primer used but is generally inferior to catalyzed epoxies.

Data are given on exposure tests involving hydrochloric acid, salt fog, caustic and four organic solvents.

Hydrochloric Acid: Figure 3 illustrates typical performance of two commercial two-package urethanes compared to a typical vinyl and epoxy-amine maintenance system. All are three-coat systems exposed on steel test panels in a wet hydrochloric acid vapor test cabinet. This severe test exposure caused general chemical attack on a three-coat, fume resistant alkyd paint in 10 days. The systems were removed from test at comparable stages of failure.

The test panels were sandblasted to a commercial finish; coating was applied by spray. Final drying time before exposure was 15 days at 70 to 75 F. A T-shaped scratch for determining undercutting was made just before exposure. Exposure was in stagnant air saturated with 20 percent hydrochloric acid vapors at 75 F.

Identification of the panels in Figure 3 is as follows: A730: three-coat commercial urethane system with phenolic-oil primer, exposed for 9 weeks. Total film thickness on plane surfaces was 5 mils. A672: three-coat commercial urethane system (catalyzed epoxy primer) with a total thickness of 5.5 mils, exposed for 9 weeks. A578: a commercial three-coat vinyl system with total thickness of 6 mils, exposed for 13 weeks. A681: a commercial three-coat, epoxy-amine system with total thickness of 6 mils, exposed for 18 weeks.

Both urethanes show the characteristic tightly cured film with excellent resistance to general penetration. They are failing at the sharp edges of the panels because the film is only 2 or 3 mils thick over the edges. Low film build on edges and prominences is a characteristic deficiency of most urethane maintenance coatings as with low-solids catalyzed epoxies. In this case, the higher film build of the vinyl and epoxy systems protected the edges longer than the urethanes.

Salt Fog: Figure 4 shows the result of exposing two typical commercial urethanes with reference systems in a salt fog cabinet to a comparable amount of edge failure. The exposure was continous fog of synthetic sea water at 110 F. Surface preparation was commercial grade sandblast with the coating spray applied. Final drying time before exposure was 15 days at 70 to 75 F. A T-shaped scratch was made on the panel just before exposure.

Panel identifications are as follows: (See Figure 4) A626: three-coat urethane system with phenolic-oil primer, exposed 9 weeks. Total thickness was 5 mils. A700: three-coat urethane system with vinyl primer, exposed for 9 weeks. Total thickness was 5 mils. 10542: three-coat epoxy-amine system exposed for 21 weeks. Total thickness was 6 to 7 mils. A589: three-coat system exposed for 18 weeks. Total thickness was 6 mils.

The urethane equalled the vinyl and epoxy-amine systems in resistance to general penetration and undercutting corrosion but showed the initiation of film failure over edges in about half the time of the reference systems.

Caustic: Commercial two-packaged urethanes did not compare favorably with the three-coat, epoxy-amine systems when exposed in a caustic test cabinet. The exposure was a fog of 20 percent sodium hydroxide solution alternated with water spray at 100 F. Of six urethane systems tested, three showed general penetration and attack on the topcoats within seven weeks and two showed complete loss of bond because of primer saponification within five weeks. The sixth urethane (over an epoxy primer) rated about the same as the epoxy-amine and vinyl systems which had no failure after nine weeks.

Organic Solvents: The typical two-package urethane coatings are outstanding in organic solvent resistance and in some cases perform better than any other air-dry coating. Figures 5 through 8 show averaged results of organic exposures of two commercial urethanes compared to two typical commercial epoxy-amine coatings. All are three-coat systems, at 5 to 6 mils total thickness; all cured at 75 F for 10 days before exposure.

Epoxy-amines were chosen as the reference type because of their general use as maintenance coatings in organic exposures.

The tests were run by immersing coated panels in the organic chemical and removing each panel at regular intervals. Immediately after removal, a marked, one-square inch of the coating surface was scraped gently with a blunt metal spatula to remove any soft or deteriorated coating. (A standard scraping procedure was used with a total of 40 passes of the spatula across the marked area.) After the scraped area was wiped with a dry tissue, thickness of the remaining film in the marked area was measured with an electromagnetic thickness gauge. The panel was returned to the test solution within 40 seconds.

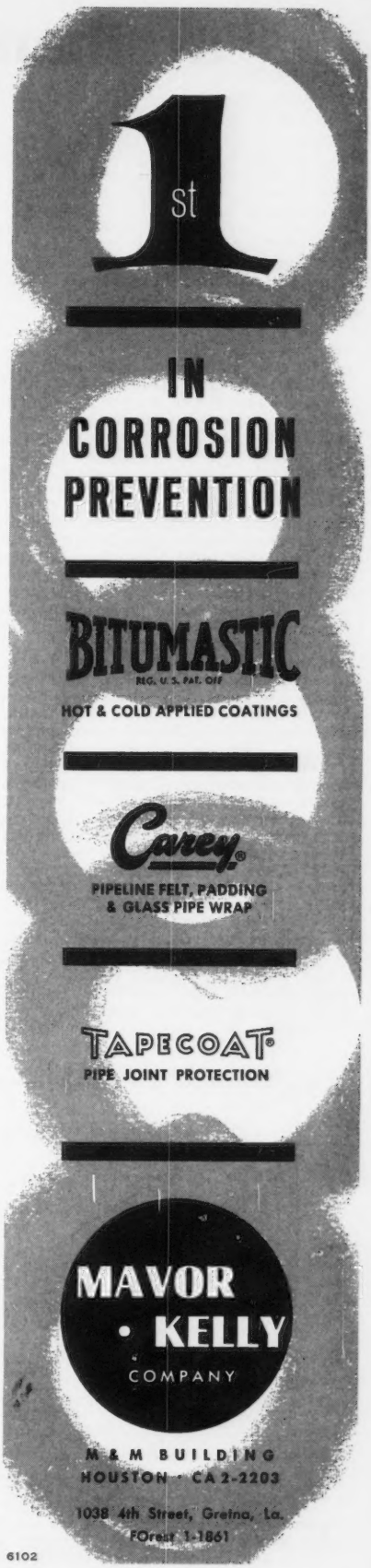
Duplicate tests were run by the same method and also by a second method, in which four panels of each system were immersed in the same chemical at the same time. One panel was removed at each interval and quickly scraped as in the first method but was not returned to the test chemical. This noninterrupted immersion proved about 30 percent more severe than the first, or interrupted, immersion test. The accuracy of the thickness measurements was estimated to be within 30 percent.

Non-immersed standard panels of each system showed no loss in thickness when scraped and measured as in the above test method.

The graphs in Figures 5 through 8 show the averaged results of the four panels (two systems and two duplicates) of each generic type. Results of the two systems within each type agreed closely.

The four organic solvents used in the test were trichloroethylene, methyl isobutyl ketone, tricresyl phosphate and dimethyl formamide. These four are discussed below.

(Continued on Page 32)



urethane coatings—

(Continued From Page 31)

The trichloroethylene (Figure 5) test was run at 75 F, and panels were removed after one minute immersions. The effect on the epoxy-amine systems was a steady softening with no film deterioration. Because trichloroethylene is highly volatile, the epoxy amines recovered film hardness within five minutes after removal from the solvent. The urethanes were not softened or noticeably affected.

The methyl isobutyl ketone (Figure 6) tests were run at 75 F, and the panels were removed after 2 minute immersion intervals. The epoxy-amines were slowly softened but recovered film hardness within 10 minutes after the end of the test. The urethanes were not affected.

Tricresyl phosphate (Figure 7), a common plasticizer and ingredient of hydraulic fluids, has extremely low volatility so that it remains in contact with a painted surface for days or weeks following a spill or leak. Because coatings on hydraulic equipment are sometimes exposed to leaks of hot tricresyl phosphate fluids, the immersion tests were run at 150 F. Panels were removed at 6-hour intervals.

The urethane systems remained unaffected through the 24-hour test. Both epoxy-amines were softened and penetrated nearly through the film within 24 hours and had not recovered normal hardness one week after the test.

Dimethyl formamide (Figure 8), used in the manufacture of several synthetic fibers, is one of the strongest solvents known for organic polymers. The test was run at 75 F, and the panels removed after 30-second immersion intervals. After two minutes, the epoxy-amines had been penetrated down to steel and had lost adhesion. The film was soft and disintegrating rapidly. There was no recovery after drying.

The urethane systems were softened about half way through the film after four minutes. The coatings retained good appearance, but disintegration of the surface was starting. After drying 24 hours, the coating had regained about 75 percent of its original toughness.

These organic solvent tests were intended to compare typical urethanes with a coating type whose good organic resistance is accepted. The tests also show the better resistance of urethanes to organics as compared to vinyls, chlorinated rubbers, alkyds, neoprenes, Hypalons and acrylics, all of which are softened quickly or dissolved by strong organics like tricresyl phospate.

Film Flexibility Retention

Coatings too often are selected for difficult service on basis of their original impact, abrasion of flexibility properties, measured within a few days after the film has cured, with little or no knowledge of how these properties will change as the film ages. The fact is now becoming more commonly recognized that all coatings, particularly catalyzed coatings, embrittle on aging. In two years of weathering exposure, impact resistance of an epoxy-amine or a polyester often is reduced to less than 20 percent of its original high value. Impact resistance of an epoxy-polyamide may be reduced as much as 40 percent. In service where weathering is combined with movement or stresses such as tank car exteriors or outdoor piping with frequent temperature changes, film failure of catalyzed epoxies often occurs in the second or third year by stress-cracking. Urethanes should be considered for these and similar applications.

The effects of ultra-violet exposure and thermal shock on commercial urethanes and epoxy-amine coatings are compared in Figure 9.

The test was run in a twin-arc cabinet, operated in cycles of 17 minutes dry ultra-violet light (black surface temperature of 160 F) and 3 minutes cold water spray. Systems tested were three commercial two-package urethanes, three coats, 4.5 to 5.5 mils total thickness and two commercial epoxy-amine systems, three coats, 5 to 6 mils total thickness.

The substrate was 24 gauge cold-rolled steel sandblasted lightly. At intervals, panels were removed, cooled to 75 F and bent over a conical mandrel to measure film flexibility. The mandrel diameter at which the film first showed a crack in a 180-degree bend was recorded. Averages of results for each generic type were plotted against exposure time (Figure 9).

All systems could be flexed without damage at the start of the test. After 150 hours' exposure which correlates to about five months of Midwest weathering, the epoxy systems had lost nearly all their original film extensibility. Three hundred hours' exposure was required to embrittle urethanes to the same point.

Two-package urethanes range from rigid to elastomeric in initial properties, varying chiefly with the type of polyol crosslinker used. The important part is that, at any point in this flexibility range, the properly formulated urethane retains its original flexibility and toughness better than any other air-drying thermosetting coatings. This property promises longer life with urethanes in outdoor applications where a combination of toughness and impact resistance or thermal shock resistance is needed.

Bonding

Two-package urethanes have fair adhesion to many surfaces but have excellent adhesion to surfaces where chemical bonding can be done, as to alkyds, epoxy coatings and wood. In these cases, the isocynate crosslinks with hydroxyl groups in the substrate. Performance on steel is improved by use of a primer, generally an epoxy or phenolic-oil. The best performing urethane maintenance systems have primers that build 2 mils or more over edges, thus counteracting the poor edge build of the urethane topcoats.

Performance of urethane maintenance systems varies in any exposure with the type of primer used. It is wise to give the coatings manufacturer details of the surface and service and to follow his primer recommendation.

Compatibility With Existing Paints

Because two-package urethanes develop a strong chemical bond to oil-containing paints, urethanes are useful for overcoating alkyd and phenolic-oil maintenance paints to up-grade the

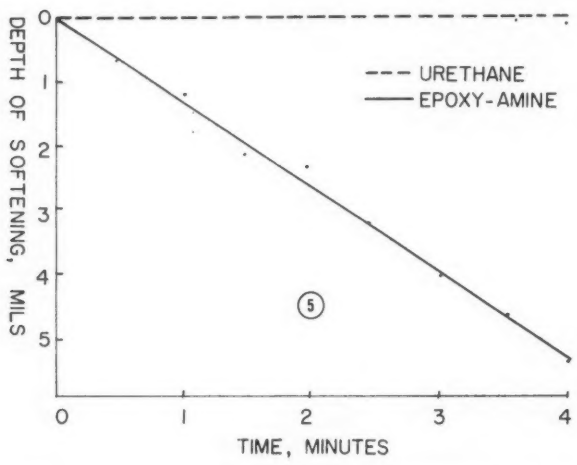


Figure 5—Comparative attack on coatings during immersion in trichloro ethylene at 75 F. Plots are averaged results of two commercial urethane and two commercial epoxy-amine systems.

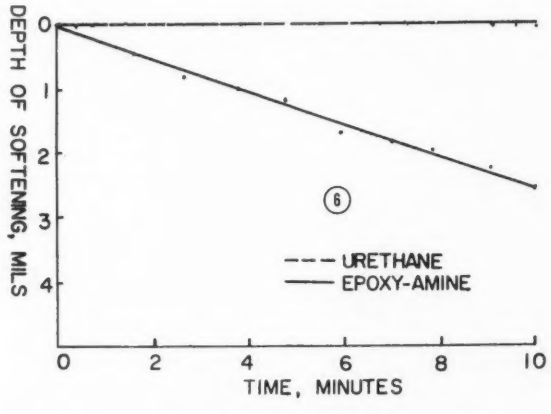


Figure 6—Comparative attack on coatings during immersion in methyl isobutyl ketone at 75 F. Plots are averaged results of two commercial urethane and two commercial epoxy-amine systems.

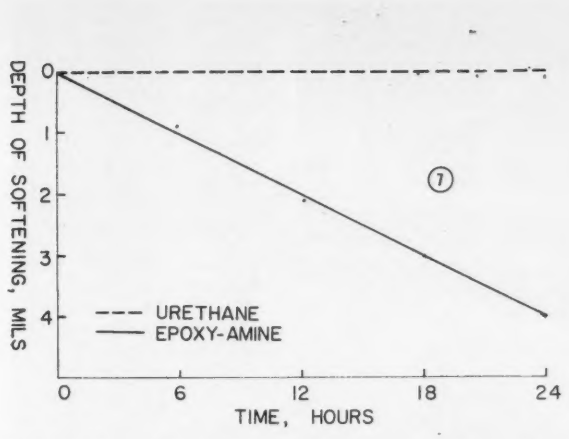


Figure 7—Comparative attack on coatings during immersion in tricresyl phosphate at 150 F. Plots are averaged results of two commercial urethane and two commercial epoxy-amine systems.

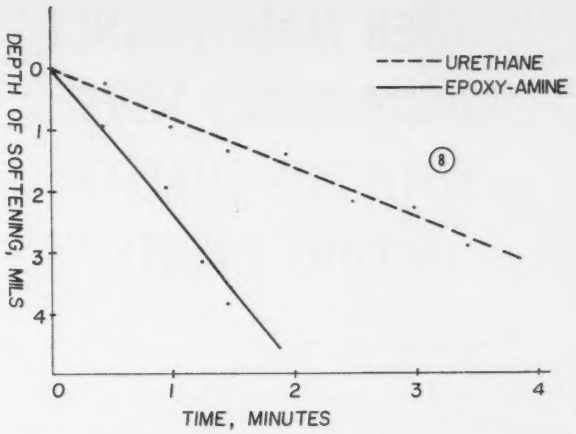


Figure 8—Comparative attack on coatings during immersion in dimethyl formamide at 75 F. Plots are averaged results of two commercial urethane and two commercial epoxy-amine systems.

chemical resistance of the paint systems. In general, catalyzed urethanes are more versatile in use over tight oleoresinous paints than are catalyzed epoxies. The chief reasons are (1) epoxies depend on mechanical bond alone, but urethanes develop chemical bond to the enamel and (2) urethane embrittles more slowly than epoxy in outdoor exposures and thus puts less stress on the softer paint underneath.

Because all thermosetting coatings, including catalyzed epoxies and urethanes, have limitations in their use over oil paints or alkyds, caution should be used. The best advice again is to consult the manufacturer and to get a recommendation based on proven field experience.

New Types of Urethanes

The performance of two-package urethane maintenance systems will be improved through use of high-build formulations so that a three-coat system will give at least six mils dry film thickness on new steel. A few high-build intermediate coat formulas are on the market.

Elastomeric urethane coatings have been laboratory tested that retain at least 100 percent elongation after aging and that air-cure to a film with good water and chemical fume resistance. Coatings of this type probably will be available for field use within two years.

Lining systems that combine the efficient thermal insulation of urethane foams with the moisture and solvent resistance of two-package urethane coatings are expected on the market within three years. They will be intended for use in fuel, solvent and process liquor tanks which require an interior lining to prevent contamination of the product and to provide insulation.

Conclusions

Two-package, air-drying urethane coatings are a useful material for heavy-duty maintenance coatings according to the tests reported in this article. The best formulations compare favorably with vinyl and epoxy coatings for protecting plane surfaces (as tank exteriors) in acid fume and wet salt exposures indoors or outdoors. The typical urethanes have low film build-over edges and often will fail earlier than vinyls or epoxies at these edges. In exposure involving organic chemicals, urethane maintenance coatings show good resistance.

The tests described above show that the typical two-package coating is more resistant to softening by high-solvency organics than are amine-cured epoxies and retains film flexibility longer than amine-cured epoxies.

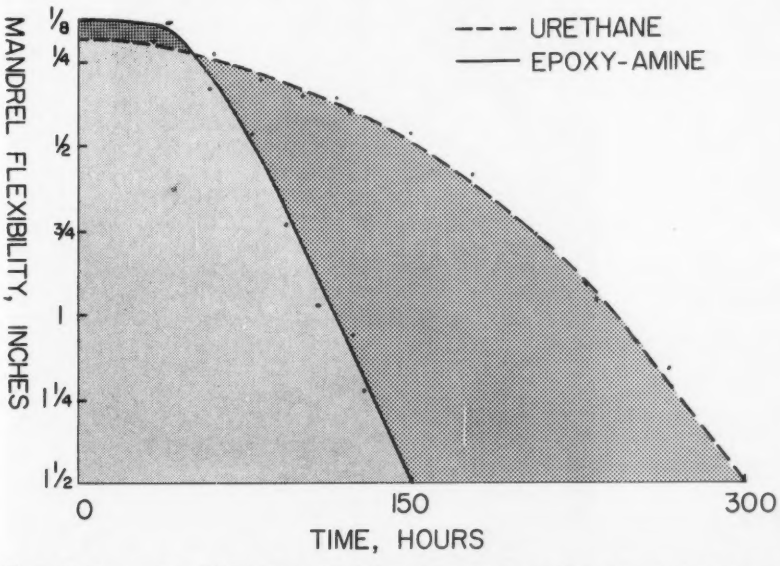


Figure 9—Comparative embrittlement of coating systems during accelerated weathering exposure in twin-arc cabinet operated at cycles of 17 minutes ultra violet light at 150 F plus 3 minutes cold water spray. Flexibility rating is mandrel diameter at which coating on 24 gauge steel panel first cracks.

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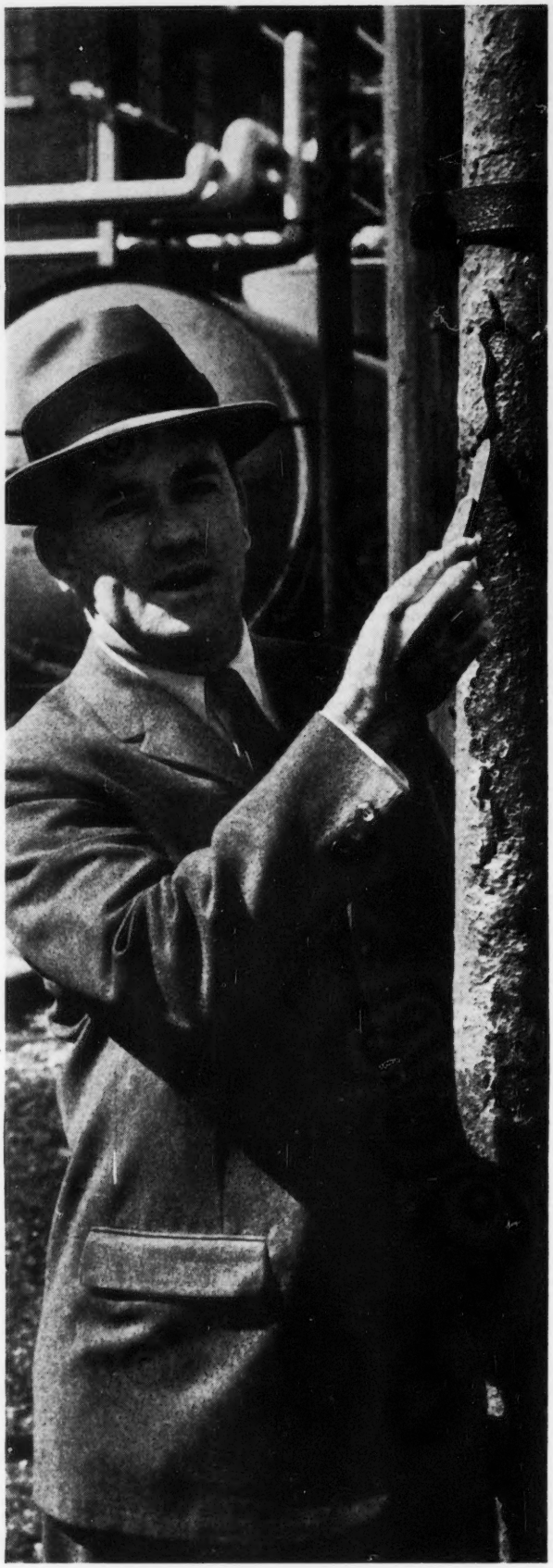

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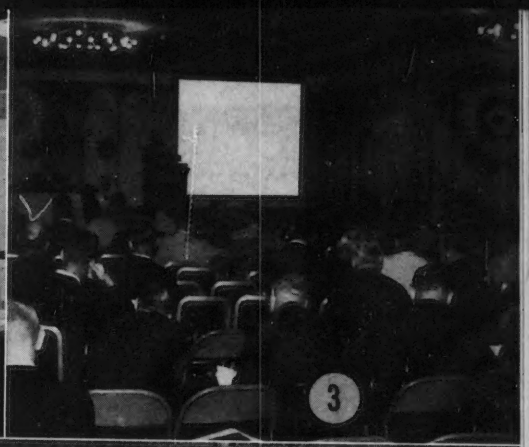

961 Attend Houston Meeting At Shamrock October 23-26

Registfation for the October 23-26 South Central Region Conference held in Houston almost reached the 1,000 mark. Total registration for the conference and 1961 Exhibition at the Shamrock-Hilton Hotel was 961.

The technical program included 9 symposia: aluminum, oil and gas production, utilities, industrial maintenance painting, pipe line, chemical and petrochemical industry, marine and offshore, cathodic protection and galvanic anodes.

The conference also included 26 meetings of NACE Technical Committees, including the NACE Research Committee.

A total of 130 ladies registered to participate in the special activities planned for them which included luncheon, a boat trip through the Houston ship channel and other tours of interest.

Fifty-five booths were set up for the exhibition held in conjunction with the 15th Annual South Central Region Conference. Exhibitors stated that they considered the exhibition to be a great success.

Spalding Memorial Award Given

H. E. Waldrip, supervisor of the Production Unit, Technical Service Laboratory of Gulf Oil Company in Houston, was presented the first J. C. Spalding, Jr., Memorial Award.

Established by the South Central Region in memory of the Late J. C. Spalding, Jr., former chairman and director representing the region, the award was made in recognition of outstanding activity in NACE by a member of the South Central Region. (See accompanying photograph.)

President and Mrs. Greco Attend

NACE President E. C. Greco of United Gas Corp., Shreveport, La., attended the Houston conference. He was interviewed twice by local television stations as part of the publicity for the conference. (See accompanying photograph.) Mrs. Greco also attended the ladies entertainment and activites during the meeting.

SOUTH CENTRAL SNAPSHOTS

Photograph 1—During the banquet, H. E. Waldrip was presented the first J. C. Spalding, Jr., Memorial Award for his outstanding work and activity in NACE and the South Central Region. The award was made by Dan H. Carpenter of Atlas Chemical Industries, Inc., Oklahoma City, Okla., chairman of the region. Photograph 2—NACE's new booth was shown for the first time as one of the exhibits at the exhibition held in conjunction with the conference. Durwood Levy of the NACE Central Office staff, was busy answering questions concerning NACE and its many publications available. Photograph 3—Attendance was good at all the symposia during which technical papers were presented on corrosion control problems of interest to engineers in the region. Photograph 4—All the booths at the exhibition were staffed with technical personnel who answered questions and explained some of the equipment and processes which were displayed. Photograph 5—NACE President E. C. Greco was asked to appear on a local television station to discuss the corrosion problems of interest to the general public.

Future Conference Dates

The 16th Annual South Central Region Conference will be held October 16-19, 1962, in San Antonio, Texas. The 1963 meeting will be held in Oklahoma City, Okla., and the 1964 conference in Dallas, Texas.

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South Central Region

GOOD ATTENDANCE at South Central Region Board of Trustees' luncheon.

Greco Tells Trustees Local Activity Way To Get New Members

A concentrated drive to enlist new members and organize new sections in South Central Region is contemplated for the coming year. Aims of this region of NACE were outlined by several speakers at the October 25 meeting of the region's board of trustees during its 1961 conference. The conference and exhibition were held at the Shamrock Hilton Hotel, October 23-26.

Reports were heard also on activity leading to the formation of new sections in the region. These reports included comments by Carl Locke, Continental Oil Co., Ponca City, Okla., concerning the possibility a section could be organized there; others pointed to work underway to determine the need for sections at Enid, Okla.; Hobbs, New Mexico; Wichita Falls and Abilene, Texas; and in Arkansas. Dan H. Carpenter, Atlas Chemical Industries, Inc., Oklahoma City, regional chairman, cited in his report the fact that the region has been static with 16 sections for the past several years.

Greco Urges Local Activity

E. C. Greco, United Gas Co., Shreveport, NACE president, urged activity at the section level to secure additional members for the association.

Carl M. Thorn, Southwestern Bell Telephone Co., San Antonio regional vice-chairman, reported on work being done on the region operating manual and on formation of a speakers' bureau. He indicated more cooperation was needed from sections if a list of speakers was to be compiled.

J. A. Caldwell, Humble Oil & Refining Co., Houston, regional director called for quickened activity in several areas to improve NACE effectiveness. His suggestions, as developed by a publicity and membership committee of which he is chairman included adequate advance publicity for regional conferences to all media; news release by sections on speakers via Central Office; urging speakers to use news media available to them such as company public relations setups and periodicals; and special notices to hometown newspapers of men elected to office or who receive other honors. He said nine sections report getting significant newspaper publicity.

Mr. Caldwell urged members to notify membership chairmen in their sections about transfers, new prospects and college graduates coming into or contemplating corrosion work.

Progress at San Antonio

Charles W. Tipps, City Public Service Board, San Antonio said that all committee chairmen have been named for the 1962 regional meeting at San Antonio October 16-19. A tentative budget figure was announced.

Frank B. Burns, General Asphalt, Inc., Wynnewood reported that work was underway for reservations and preliminaries on the 1963 Oklahoma City conference.

Morris Bock, Sun Oil Co., Dallas, was announced as general chairman of the scheduled Dallas 1964 regional meeting.

Shreveport Elects Officers for 1962

The Shreveport Section has elected its 1962 officers. D. A. Tefankjian of Texas Eastern Transmission Corporation, Shreveport, is the new chairman.

John W. Graves of United Gas Pipe Line Company, Shreveport, is vice chairman. Secretary is W. E. Moore of Tretolite Company, Haughton. Treassurer is J. A. Irvin of Arkansas Louisiana Gas Company, Shreveport, and Ned C. Stearns of Irish Engineering Service, Inc., Shreveport, is trustee.

For its meeting January 2 in the Petroleum Club of Shreveport, the section has scheduled Barnard P. Goodman of Tube-Kote, Inc., Houston, to give an illustrated talk entitled Economics of Coating for Corrosion and Paraffin Prevention in Oil Well Tubing.

On November 7, the section heard Carl E. Locke of Continental Oil Company speak on the Anatrol System of Corrosion Control.

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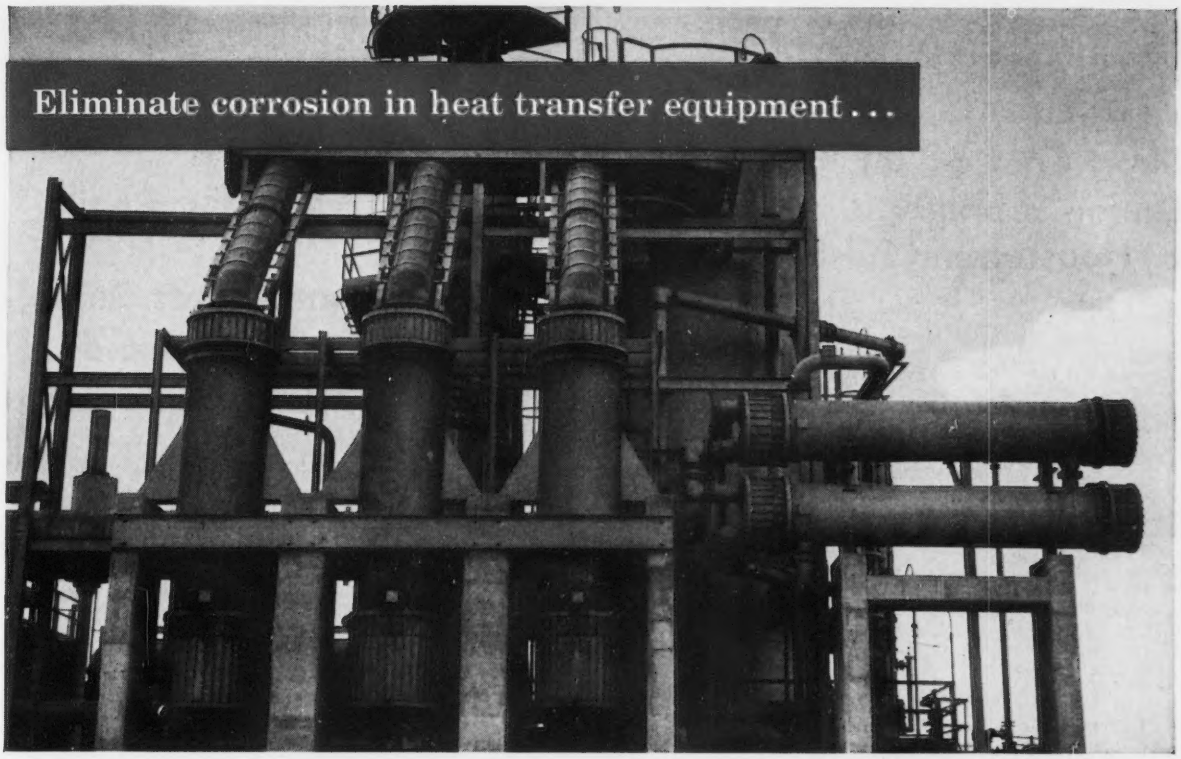

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Components of all sizes of "Karbate" heat exchangers are manufactured in quantity and carried in stock. You save money . . . you save time . . . when you specify "Karbate" impervious graphite shell and tube heat exchange units.

Series	Nom. Dia. Shell	No. of 7/8" I.D. x 1¼" O.D. Tubes	HEAT TRANSFER SURFACE OUTSIDE TUBES, SQ. FT.				
			6' Long Tubes	9' Long Tubes	12' Long Tubes	14' Long Tubes	16' Long Tubes
90	6"	9	17.7	26.5	35.4	—	—
190	8"	19	37.3	55.9	74.5	87.0	99.5
310	10"	31	45.6*	91.2	121.6	141.9	162.2
420	14"	42	81.7	123.3	164.8	192	220
640	16"	64	124.3	187.7	251	293	335
850	18"	85	165.2	250	334	389	444
1090	20"	109	213	320	427	499	570
1350	22"	135	263	397	530	618	706
1630	24"	163	317	479	640	746	852
1950	26"	195	379	573	766	892	1017
2330	28"	233	453	684	914	1065	1217
2680	30"	268	521	786	1051	1225	1400
3490	34"	349	679	1024	1368	1595	1822
4390	38"	439	855	1289	1722	2009	2295
5960	42"	596	1162	1749	2336	2727	3118
6850	45"	685	1335	2010	2685	3135	3585

***This unit has 4½' long tubes.**

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UNION CARBIDE

South Central Region

(Continued From Page 36)

Short Courses Set During February By Houston and Tulsa

Two South Central Region sections, Houston and Tulsa, have scheduled corrosion control short courses for February. Dates, locations and some of the subjects to be covered are given below. As soon as the programs have been completely worked out, they will be published in CORROSION.

Houston Short Course

Focusing on corrosion control fundamentals and field practices in oil and gas production, transmission pipelines, utilities, refineries and chemical plants and commercial and residential construction, the Houston Section's 8th Annual Short Course will be held February 1-2 at the Rice Hotel in Houston. D. R. Fincher of Tidewater Oil Company is chairman.

Chairmen to head the various sessions are as follows:

M. E. Frank of Tennessee Gas Pipeline Company, corrosion control in transmission pipeline systems; G. R. Roberson of Standard Oil Company of Texas, corrosion in oil and gas production; E. C. Winegartner of Humble Oil & Refining Company, corrosion in refineries and chemical plants; C. L. Mercer of Southwestern Bell Telephone Company, corrosion in utilities, and M. A. Riordan of Rio Engineering Company, corrosion in commercial and residential construction.

Tulsa Short Course

To stress control of pipeline corrosion, Tulsa Section's 12th Annual Corrosion Short Course is recommended especially for foremen, superintendents, design engineers, field engineers and inspectors. It will be held February 21-23 at the Mayo Hotel, Tulsa. Chairman is E. W. Lawlor, consultant.

Subjects to be covered include pipeline coatings, corrosion surveys, cathodic protection, internal pipeline corrosion protection and instrumentation.

Sabine-Neches Elects Hugh S. Hickerson 1962 Chairman

The Sabine-Neches Section has elected its 1962 officers. Chairman is Hugh S. Hickerson, Jr., of Pittsburgh Plate Glass Company, Chemical Division, Lake Charles, La. Vice Chairman is Hal J. Pitman of Southern Union Gas Company, Port Arthur, Texas, and Secretary-Treasurer is Carl D. Watson of Nalco Chemical Company, Beaumont, Texas.

On November 2, the section heard R. E. Latham and Roy Davis, both of Gray Tool Company, Houston, Texas, discuss the manufacture, fabrication and performance of bi-metallic pipe. This meeting was held at Orange, Texas, and was attended by 15 persons.

•

Teche Section heard John Broussard, Lafayette Parish Civil Defense director, and Jerome Romagosa, radiologist, discuss nuclear radioactive fallout and community survival at its November 1 meeting.

•

East Texas Section scheduled William C. Koger of Cities Service Petroleum Company, Bartlesville, Okla., to speak November 28 at Hotel Longview in Longview.

•

Alamo Section met with the Communications Engineers at the Officers' Club, Randolph Air Force Base, on October 19 to hear Jim Sage, science demonstrator for Southwestern Bell Telephone Company, discuss Voices Across Time and Space dealing with projects under way to expand worldwide telephone service and to possibly provide worldwide television transmission by bouncing signals off a series of man-made satellites.

Canadian Region

Canada Sets Dates For Toronto and Calgary Conferences

Dates have been set for Canadian Region's 1962 meetings. Eastern Division will hold its sessions at the King Edward Hotel, Toronto, January 17-19. Harold A. Webster, Corrosion Service Ltd., is conference chairman.

Western Division will hold its sessions at Calgary, Alberta on February 7-9. J. U. Messenger, Mobil Oil of Canada, Ltd., Calgary is technical program chairman.

•

Edmonton Section heard A. Pawlinchuk of Alberta Phoenix Tube Mills discuss methods of manufacturing pipe on September 28. He particularly discussed his company's method and related phases such as quality control and non-destructive testing. After his talk, he conducted the section on a tour of the Alberta Phoenix Tube Mills plant. Twenty-eight persons attended the meeting.

Write on company letterhead for complete information, literature and recommendations.

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MARKAL "D-A" COATINGS— provide metals a permanent "shield" against corrosive weather conditions, salt sprays, sulfur fumes from burning coal, water steam, hot acids, mild alkalis or other corrosive conditions at ambient temperatures up to 600°.

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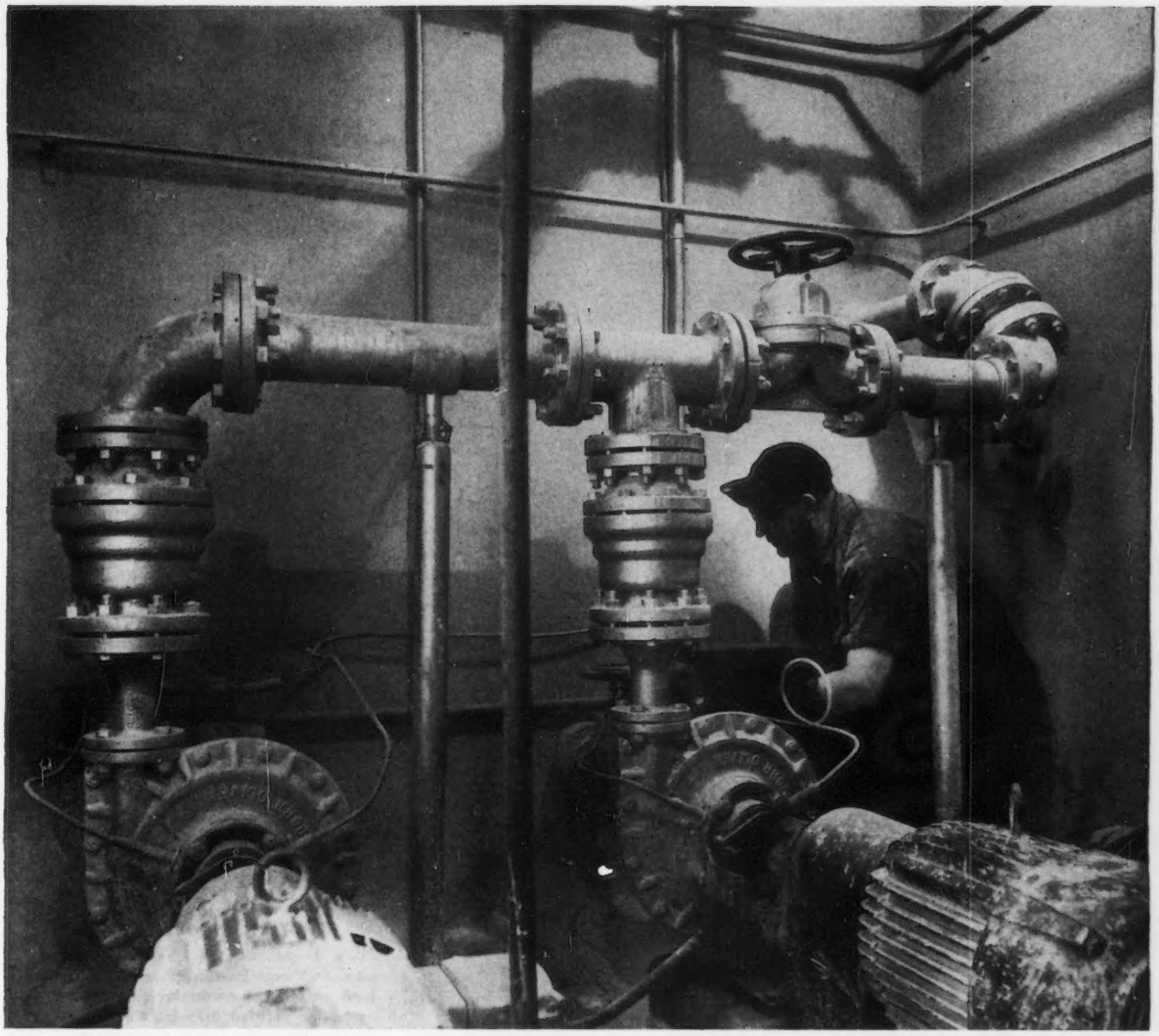

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SARAN LINED PIPE—no failure in nine years carrying 15,000,000 gallons of hot acid!

When a process uses hundreds of gallons a minute of hot corrosive acids . . . including concentrated hydrofluoric . . . trouble-free production depends on the corrosion resistance of piping. The Saran Lined Pipe shown above keeps its hot acid cargo flowing, year-in, year-out, without failure.

At the Latrobe, Pa., plant of Kennametal, Inc., hundreds of feet of Saran Lined Pipe and valves carry concentrated hydrochloric (20° Baumé) and hydrofluoric (70%) acids, as well as acidic metallic salts, between processing stations. Solution temperatures reach as high as 150° F., with some sections of the pipeline exposed to outdoor temperatures. Says Robert R. Nagel, Manager of Metallurgical Operations, "Saran Lined Pipe gives a permanent installation, with less maintenance than any other pipe we've tested. For the past nine years, we've had no failures at all, even where there's vibration from pumps in the line.

"Saran Lined Pipe has a strong steel casing that supports itself. It won't crack from accidental blows, an important safety factor for our operating personnel, considering the corrosive materials we handle. We even use Saran Lined valves with other pipe systems because we consider them leak-proof."

For dependable piping systems that will stand up under even the most corrosive of chemicals, consider Saran Lined Pipe. Saran Lined Pipe, fittings, valves and pumps are available for systems operating from vacuum to 300 psi, from below zero to 200° F. They can be cut, fitted and modified easily in the field without special equipment. For more information, write Saran Lined Pipe Company, 2415 Burdette Avenue, Ferndale, Michigan, Dept. 1565AU12.

THE DOW CHEMICAL COMPANY DOW Midland, Michigan

Western Region

204 From 16 States Register at Portland Conference Oct. 3-5

Attendance totaled 204 at the Eleventh Annual Western Region Conference in Portland, Oregon, October 3-5. Of the 16 states represented, Oregon led with 71; following were Washington with 59 and California with 40.

Planned so as to interest professional engineers and architects as well as corrosion engineers, the technical program consisted of eight symposia covering almost all facets of corrosion and controls. The symposia were corrosion resistance and application of new materials, corrosion principles, cathodic protection, corrosion of building and related mechanical equipment, fresh water corrosion, marine corrosion and protective coatings, utilities, and corrosion in the pulp and paper industry.

Portland's mayor, Terry D. Schrunk, opened the conference with a welcoming address. Also attending the conference were NACE President Edward C. Greco, Vice President Rolland McFarland, Jr., Treasurer C. G. Gribble and Executive Secretary T. J. Hull.

J. R. Smith, publicity and printing chairman, reports all local Portland newspapers carried articles on the meeting. It also was announced on all local radio stations and a local TV station.

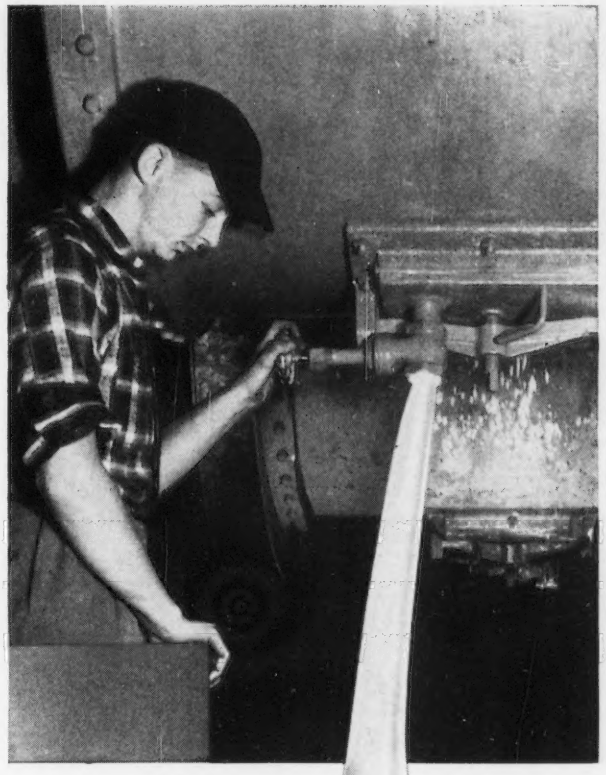
The 1962 Western Region Conference will be held in October in San Diego, Cal.

PHOTOGRAPHED AT PORTLAND

Photo 1—At banquet, Charles Seavey of Pacific Northwest Bell Telephone Company, Portland, presents an entertaining electronics demonstration entitled Breaking Through the Hay Barrier. Photo 2—NACE President Edward C. Greco injects an amusing note while speaking at the banquet. Enjoying his remarks are (left to right) Arrangements Chairman W. H. Kipp, Jr.; NACE Executive Secretary T. J. Hull; Western Region Director E. F. Bladholm and Western Region Chairman O. L. Hudrlik. Photo 3—Conference Chairman Don D. McGregor of U. S. Corps of Engineers, Portland, addresses the some 250 attending the banquet in the Crystal Ballroom of the Benson Hotel October 5. To his right is the Western Region's Secretary-Treasurer, S. F. Grubb of Portco Corporation. Photo 4—Arrangements Chairman W. H. Kipp, Jr., of Waterworks Supplies Company, Portland, speaks at the banquet. Photo 5—Program Chairman John Van Bladeren of Northwest Natural Gas Company, Portland, presents a paper dealing with corrosion problems in hot water tanks.

(Continued on Page 42)

See Page 53 for Preview of 1962 NACE Conference March 19-23, Kansas City's Municipal Auditorium



Another Major Development by U.S. Stoneware Research

A vinyl-based protective coating developed especially to simplify maintenance painting.

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These newer Tygon Coatings offer more protection per dollar

Series "AV"—Tygon Airless Vinyl for maximum corrosion-resistance at lowest applied cost.

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Tygon Super-Build offers two important advantages in maintenance painting programs: (1) excellent resistance to chemical attack and (2) a 3-mil thick (dry) film obtained in a single cross-coat application.

It can be used just as it comes from the can — no thinning required. And it dries to touch in less than 15 minutes. Calculated coverage better than 450 square feet per mil thickness per gallon. Applies over Tygorust or any Tygon Primer.

Series "SB" exhibits the same high resistance to corrosive attack characteristic of all Tygon protective coatings. Series "SB" is resistant to an extremely wide range of acids, alkalies, oils, greases and alcohol at atmospheric temperatures ranging up to 220° F.

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Western Region

(Continued From Page 40)

Officers Elected For 1962 Conference In San Diego, Cal.

The Western Region has selected its 1962 conference officers, with the exception of Conference Co-Chairmen and the Hospitality Chairman. This conference is scheduled for San Diego, Cal.

These officers are as follows:

Program Co-Chairmen: F. O. Waters of San Diego Utilities Department, La Mesa, Cal., and Ray Ditto of California Water & Telephone Company, Chula Vista.

Publicity Chairman: R. C. Tullis of Escondido Mutual Water Company, Escondido, Cal.

Arrangements Chairman: Albert F. Hooper, Jr., of Convair, Division of General Dynamics, San Diego, Cal.

Facilities Chairman: O. L. Hepner of Alvarada Filtration Plant, La Mesa, Calif.

Registration Chairman: Richard Spear of Pacific Telephone & Telegraph Company, San Diego, Cal.

Education and Ladies' Program Chairman: Donald P. Armbruster of San Diego Gas & Electric Company, San Diego, Cal.

Secretary-Treasurer: L. L. Flor of Helix Irrigation District, La Mesa, Cal.

New Nat'l Officers and Directors will be installed at Kansas City Conference. See Page 62

Northeast Region

ROBERT V. JELINEK, professor of chemical engineering at Syracuse University, discusses fundamentals of corrosion at Philadelphia Section's meeting October 30. Also shown is Section Chairman Douglas F. Woolley, Jr.

Philadelphia Hears Fundamentals Talk

The Philadelphia Section heard Robert V. Jelinek, professor of chemical engineering at Syracuse University, discuss fundamentals of corrosion on October 30.

This meeting was Students' Night and students from Drexel Institute of Technology, the University of Pennsylvania, Temple University and Villanova University attended. Members of the Drexel Institute of Technology Evening College Alumni Association also attended. Coffee speaker was L. Loyd Bohn, chairman of the Department of Physics at Temple University; he spoke on the use of non-destructive tests to determine the authenticity of old paintings.

Guest Speaker Jelinek is shown addressing the section and guests in the accompanying photograph. To his right is Section Chairman Douglas F. Woolley, Jr.

At its December 19 meeting, the section will elect its 1962 officers. At this meeting, G. E. Best, past president of NACE, will speak on Management's Concern With Corrosion.

The technical program has been planned through May. It is as follows:

February 6—Paul A. Ganser of Philadelphia Electric Company will speak on Gas Line Corrosion Problems in the Philadelphia Area.

April 9—S. O. Meyer of Betz Labs will discuss H_2O Corrosion—Industry's Tireless Foe.

May 31—S. C. Frye of Bethlehem Steel Company will speak on Organic Coatings.

Kanawha Valley Names 1962 Officers

The Kanawha Valley Section has elected new officers and committee chairmen for 1961-62.

W. R. Lambert of United Fuel Gas Company, Charleston, W. Va., is the new chairman. C. C. Stoneking of Hope Natural Gas Company, Clarksburg, W. Va., is vice chairman and program chairman. Russell L. Davisson of Union Carbide Chemicals Company, South Charleston, W. Va., is vice program chairman.

George Elder of Union Carbide Chemicals Company, South Charleston, is secretary. F. A. Radford of Cabot Corporation, Charleston, is treasurer and arrangements chairman.

Other chairmen are Dick Ballard of United Fuel Gas Company, Charleston, publicity; L. T. Moore of Tennessee Gas Transmission Corporation, Catlettsburg, Ky., membership; F. W. Floyd of United Fuel Gas Company, Charleston, hospitality, and J. M. Bates of Union Carbide Chemicals Company, South Charleston, nominating committee.

Composing the student interest committee are George C. Cox, consulting engineer, Charleston; L. S. Van Delinder of Union Carbide Chemicals Company, South Charleston; Rodney Schroeder of Standard Ultramarine and Color Company, Huntington, W. Va., and A. W. Horne of Union Carbide Plastics Company, Marietta, Ohio.

Set Meeting Dates

The section also has announced meeting dates and places through May. These meetings are all scheduled in West Virginia.

January 18—Charleston—Chemical
March 22—Charleston—Cathodic
May 17—Parkersburg—Chemical

On November 16 the section met in Charleston. W. E. Cook of Pipeline Service Corporation, Glenwillard, Pa., presented movies on cathodic protection, coatings and concrete coated pipe.

The section has established the practice of inviting two students and a faculty member or two faculty members and one student from a local school to attend meetings.

Also the section has voted to present certificates to future retiring chairmen and to all past chairmen.

(Continued on Page 46)

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Two-fisted Coreco protection pays for itself many times. Whether your problem is better solved by rectifiers or magnesium anodes, our engineers have the knowledge to determine lowest overall installation and operating costs. It costs you nothing to check with Coreco engineers.

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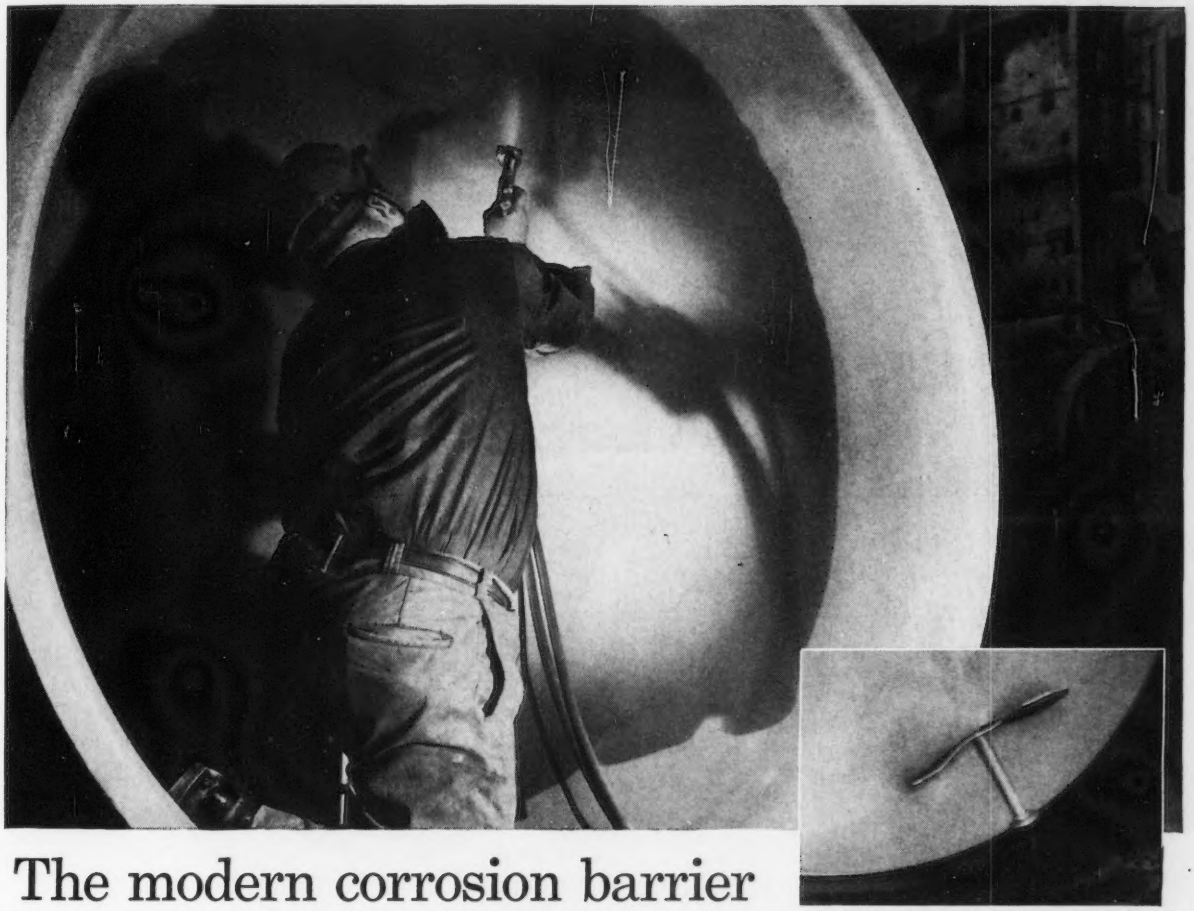
PENTON* at work: PFAUDLON 301



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Pfaudlon 301 is the name of a water-suspension coating based on Penton which provides an economical way to protect equipment from the corrosive effects of direct product contact with corrosive fluids and atmospheres.

Resistant to more than 300 different chemicals and chemical reagents Pfaudlon 301-Penton coatings are hard, nonporous, glossy and smooth. They can be readily applied as an interior or exterior coating to such metals as mild steel, cast iron, stainless steels, Hastelloys, brass, bronze or copper.

Pfaudlon 301 coatings can serve on many kinds of equipment—storage tanks, open vessels, hoods, blowers, ducts, baskets, pump and pump parts.

Pfaudlon 301-Penton coatings can be supplied by Pfaudler on new process equipment, or applied on existing equipment as a custom coating by Pfaudler-licensed applicators in your area.

Pfaudlon 301 coatings are but one of the many ways in which Penton, the modern engineering thermoplastic, is now serving industry in combating corrosion of many types. Penton processing components of all types are now readily available. Write for your copy of "The Penton Buyer's Guide," a complete listing of suppliers of valves, pipe and fittings, pumps, meters, tank linings and coated parts made with this low cost, durable, and reliable corrosion barrier.

Pfaudlon 301 Stays On: Penton-coated agitator used to mix highly abrasive sandlike slurry in 3½% of 37% HCl in H_2O shows no visible corrosion after twelve months of steady production.

Simple as ABC. Send for The ABC's of Penton for Corrosion Resistance. *This bulletin rates Penton's performance at temperatures up to 250° F. when exposed to over 300 chemicals and reagents.*

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For information on Pfaudlon 301 coatings for new or existing process equipment, write: Pfaudler Permutit, Inc., Rochester, New York.

X-TRU-COAT Electric Weld Steel Pipe protects high voltage electrical cables

Engineers for a major utility planned to run high voltage lines under a New England river. They used Republic X-TRU-COAT Plastic Coated Electric Weld Pipe to assure high strength with superior workability and corrosion resistance.

Pipe ends were flared and welded using chill rings to prevent welding beads that might damage electrical insulation on the cable. Where coating was cut back for welding, the bare steel was protected with Republic X-TRU-TAPE pressure sensitive tape and primer. The finished underwater span, 700 feet long, rests on a six-inch bed of sand and is covered with another twelve inches of sand.

Republic Electric Weld was particularly well suited for the job because this pipe has smoother inner walls, an important consideration in pulling through the very expensive and heavy conductor. Physical properties of Electric Weld Pipe are obtained by cold work rather than high carbon content, thus the steel possesses greater ductility for easier bending and flaring. The low carbon content also improves weldability.

Proved by hundreds of utilities in 50 million feet of installations, Republic X-TRU-COAT stops pipe line corrosion. High density polyethylene coating is continuously extruded over steel pipe —½″ through 8″ nominal. Pipe is undercoated with a special adhesive that bonds the plastic to the steel, eliminating underfilm migration.

The polyethylene is immune to corrosion or attack from a wide range of chemicals. Dielectric characteristics are high and remain high. For complete data, contact your nearest Republic sales office or mail the coupon.

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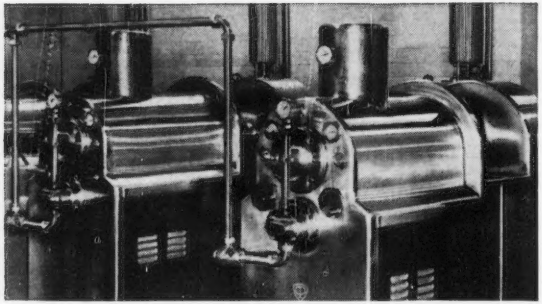
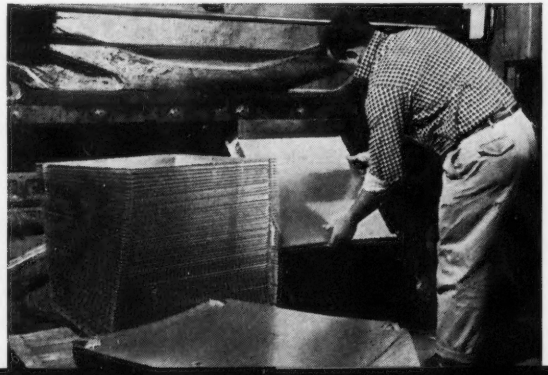
REPUBLIC STEEL



REPUBLIC ELECTRUNITE® STAINLESS STEEL TUBING provides excellent resistance to chemical action, physical damage, temperature and pressure extremes. Meets both ASTM and ASME requirements. Republic Stainless Steel Tubing is available in sizes from ¼″ through 5″ O.D. Send coupon for full information.

REPUBLIC GALVANNEALED SHEETS have a coating of graduated iron-zinc alloys between the base metal and the pure zinc outside. Coating stays tight—resists flaking, peeling, spalling. The tough, uniform protective finish is spangle free, minimizes corrosion problems, provides an ideal base for paint, withstands temperatures to 750°F.



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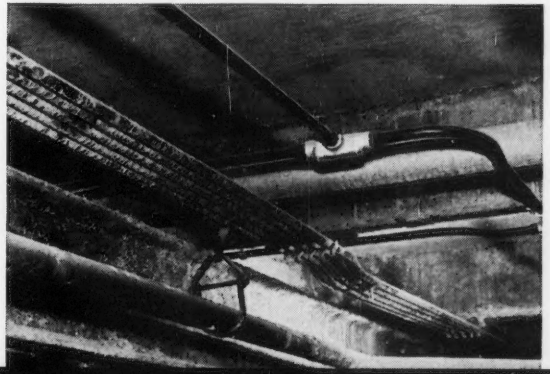
REPUBLIC ELECTRUNITE "DEKORON®-COATED" E.M.T. AND RIGID STEEL CONDUIT give maximum protection against corrosive atmospheres. Tough polyethylene encases the galvanized steel in an end-to-end armor that is impervious to most corrosive chemicals, gases, fumes, steam, and condensation. Outlasts standard conduit by ten to one or more.



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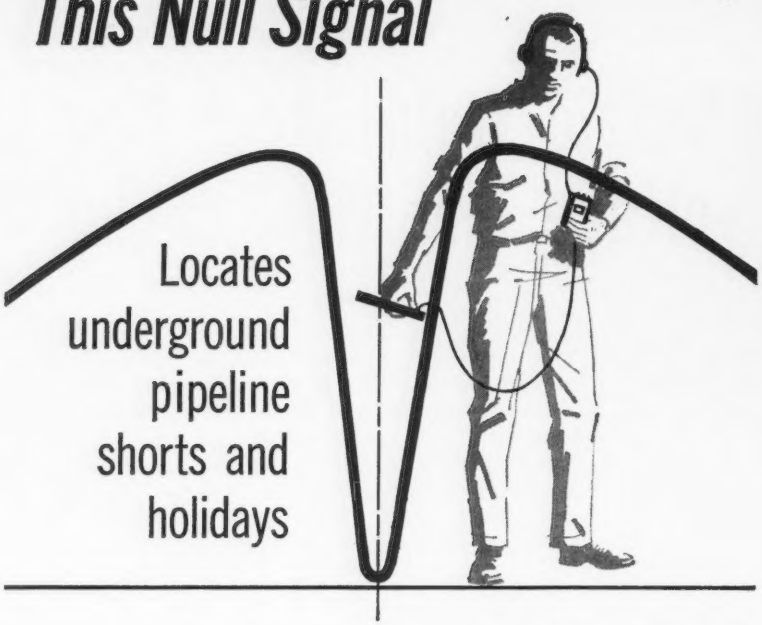
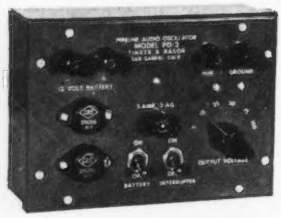
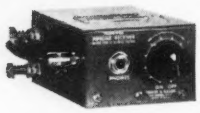
This Null Signal



Locates underground pipeline shorts and holidays





The null search system, teamed with Tinker and Rasor all-transistorized, Pearson-type Detectors, has greatly simplified the location of electrical shorts and holidays in underground pipe coatings. Findings are made within inches because the null is a positive factor which is either present or not present.

The Tinker and Rasor Underground Detector employs a transistorized oscillator which generates a 15 watt, 750 cycle, stable A.C. audio-frequency signal. Two 750 cycle resonate filters in the receiver reject unwanted signals. Using a low impedance search coil, one man can inspect a buried line at a rapid walk. Shorts will be indicated sharply and surely. A two man team is equally effective in locating holidays.

Tinker and Rasor has prepared a complete data kit which describes the null search system as well as other recommended procedures for inspecting protective coatings. Material includes technical data on equipment, general discussion of types of detectors, theory of operation, etc.

Engineering Note:
To insure a perfect application, include Tinker & Rasor Holiday Inspection. Write for specification guide.

Quality Control for Coating Application

TINKER & RASOR

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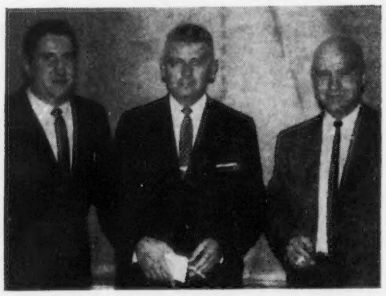
Northeast Region

(Continued From Page 42)



G. F. Bush (middle) of Ford Motor Company discussed corrosion of exterior automotive trim at Pittsburgh Section's meeting on November 2. Shown with the guest speaker are Section Chairman William Mathay (right) and Program Chairman Anthony Amurgis.

Pittsburgh Hears Talk On Auto Trim Corrosion

Eighty persons attended Pittsburgh Section's November 2 meeting to hear G. F. Bush of Ford Motor Company discuss corrosion of exterior automotive trim.

The lengthy question and answer period following the talk and the good attendance indicated the high interest in the subject, reports Program Chairman Anthony Amurgis, who is shown (left) in accompanying photograph with guest speaker Bush (middle) and Section Chairman William Mathay.

Schenectady-Albany-Troy Section Elects Officers

The Schenectady-Albany-Troy Section has elected new officers for the 1961-1962 period. The new officers are Chairman Henry Suss of Knolls Atomic Power Laboratory, General Electric Company, Vice Chairman John A. McGowan of ALCO Products, Inc., and Secretary-Treasurer Edwin C. Brush of the Research Laboratory, General Electric Company.

The section also has scheduled its program through January. On November 28, D. K. Priest of The Pfaudler Company, Rochester, N. Y., will speak on Materials Problems for the Re-entry Environment. On January 30, R. J. Enrice of Knolls Atomic Power Laboratory, General Electric Company, Schenectady, N. Y., will discuss Manufacturing Controls to Establish Corrosion Resistance of Zirconium Alloys.

DEATHS

George H. Young, director of research of Mellon Institute, Pittsburgh, Pa., and a member of NACE since 1946, died October 10. He had been active in corrosion work for about 25 years. He had served as chairman of the American Coordinating Committee on Corrosion and done consultative work for the Army and the Navy. For many years he was in charge of research fellowships on protective coatings at the institute.

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Most maintenance paints cannot be applied below 40° F. Not so with Carbo Zinc 11 *... the only protective coating that can be applied outdoors in cold weather.* Paint *anytime*, 12 months of the year, indoors or out ... keeping a *smaller* crew busy full time. You still get protection far superior to standard maintenance paints.

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Use Carbo Zinc 11 without topcoat, or as primer with vinyl, Hypalon, epoxy, or inorganic topcoat. Resists undercutting and subfilm corrosion. Low material, application and maintenance costs.

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4½ oz. Sea Rite Salt crystals in one gallon tap water produces laboratory sea water quickly, inexpensively. Provides uniform, constant results based on 77 composite analysis. Immediate delivery — any quantity. Technical literature on request. Write, wire, phone now!
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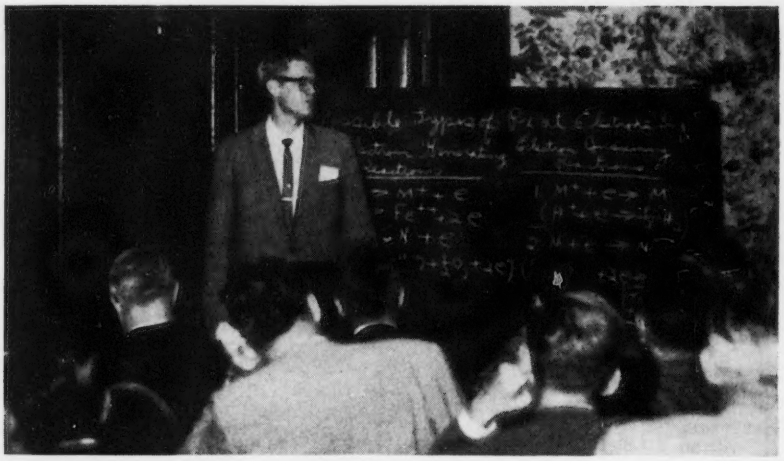
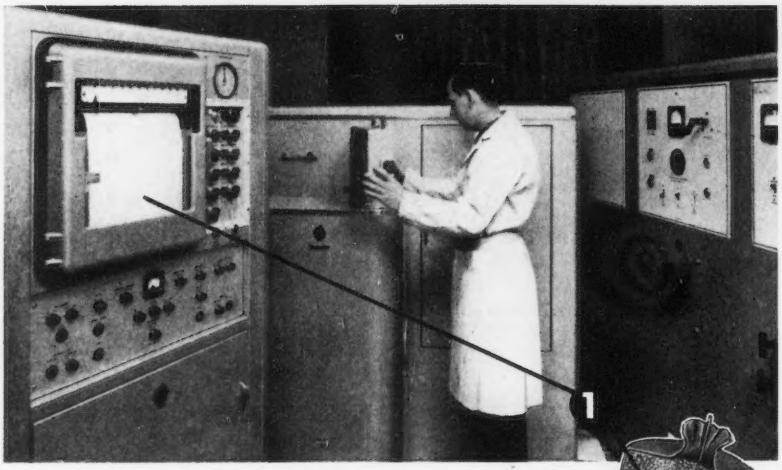




GERALD G. WILSON of the Institute of Gas Technology discusses the significance of electrode potentials in cathodic protection design before a meeting of Chicago Section October 17.

Electrode Potentials Discussed at Chicago By Gerald G. Wilson

Members of Chicago Section at their October meeting heard Gerald G. Wilson, Institute of Gas Technology, Illinois Institute of Technology, Chicago discuss "Simple Concepts of Potential—Key to Understanding of Reference Electrode Measurements and Cathodic Protection." The dinner meeting was held October 17 at Chicago Engineers' Club. Essentially, Mr. Wilson pointed out, an understanding of reference electrode data involves locating electron generating reactions as opposed to electron consuming reactions.

He told his audience that if this concept can be kept in mind when examining the data accumulated in potential surveys, it often helps in simplifying the problem and in reaching a proper solution. Te enumerated five factors in electrode potential differences in the order of their magnitude of effect: 1. Electrode composition. 2. Electrolyte composition. 3. Adsorbed films. 4. Temperature. 5. Mechanical stress.

Points of his discourse were illustrated by equations of the mathematical concepts involved.

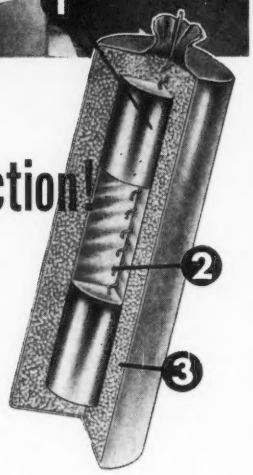
Southwestern Ohio Named Affiliate of Science Group

The Southwestern Ohio Section has been named an affiliate of the Technical and Scientific Societies Council of Cincinnati, Ohio. Representing the section on the council will be W. E. Eckert of Koppers Company, Inc., Cincinnati, and R. L. Wood of The Cincinnati Gas & Electric Company.

On October 24, the section heard William T. Woodson of St. Louis Metallizing Company speak on organic coating and tank linings.

North Central Region

add them all up . . .
They Mean Better Protection!



Standard Magnesium VIBRA-PAK anodes provide the best possible protection against corrosion on underground and underwater structures because:

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No matter what the installation, Standard Magnesium anodes will provide far better, more economical protection against corrosion. Insist on Standard Magnesium anodes.

Write us and we'll send you, in booklet form, the complete story on why Standard Magnesium Vibra-Pak anodes are better!

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Upper left: Otto H. Fenner, Monsanto Chemical Co., St. Louis, general chairman of the October 9-11 North Central Region Conference at St. Louis shares a joke with Rolland McFarland, Jr., Hills-McCanna Co., Carpentersville, Ill., president-elect of NACE. Mr. Fenner's Tyrolean hat was part of the decorations for an entertainment session featuring St. Louis beer. In the two lower photographs are pictured four of the ladies attending the conference. Upper right: Otto Fenner, chairman of NACE T-6D committee discusses with members of the committee its project for devising a system of indicating plastic materials formulation by numbers. Lower right: Father Dismas Clark, founder and executive of St. Louis "Dismas House," tells a luncheon audience about his work. Dismas House is maintained by persons of all faiths as a way station at which felons released from prison may be trained in useful jobs.

Trustees Recommend Ways to Improve Administration

Improved administration of region affairs is expected as the result of actions taken at a meeting of North Central Region's board of trustees at a Monday, October 9 luncheon in Chase Park Plaza Hotel, St. Louis. The board met during the region's annual conference featured by a technical program of 29 papers in nine symposia and six technical committee meetings.

C. W. Ambler, Jr., American Zinc, Lead & Smelting Co., regional chairman presided.

After hearing the reports of regional officers, a discussion revolving around the efficient conduct of region affairs developed into a series of recommendations, including a requirement that men selected as section trustees be required to agree in advance that they will attend the board's meetings. This decision came about because of poor representation at meetings of section trustees.

Other Regional Matters Weighed

Among the unresolved problems facing the region are the realignment of boundaries brought about by the addition of new sections and a change in the

(Continued on Page 50)

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MATERIALS
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North Central Region

St. Louis Meeting

(Continued From Page 49)

dates on which various steps in the nomination and election of officers are scheduled.

Regional Meetings Set

Reports on progress being made for the scheduled meetings of the region were discussed, especially that planned for Detroit on October 9-11, 1962. Other regional meetings are scheduled for Kansas City, 1963; Minneapolis-St. Paul, 1964. Cincinnati is to be invited to hold the 1965 meeting.

There was some discussion concerning a shift of the 1963 meeting to another place because of the close time relationship between it and the 1962 NACE Conference to be held in Kansas City next March.

Otto H. Fenner, Monsanto Chemical Co., St. Louis, general chairman of the conference reported that registration at the meeting was disappointing but that the "Exhibition in Miniature" brought funds which helped the conference committee balance its budget.

Short Course Is Planned By Twin Cities Section

Twin Cities Section (Minneapolis-St. Paul) is planning a corrosion control short course. The section also has been invited to hold the 1964 regional meeting.

Practical ideas to help you Stop CORROSION

HOW YOU SAVE MONEY BY USING "HIGH POTENTIAL" RATHER THAN "STANDARD" MAGNESIUM ANODES

Why do you use magnesium anodes to stop corrosion of pipe lines, tank bottoms, oil well casing, etc.—rather than other types of galvanic anodes that sell for less money per pound?

The answer is, of course, because magnesium gives you a lower overall cost for cathodic protection. Magnesium's higher potential (approximately 0.4 volts) makes it possible to achieve protection with fewer installations.

If the above is true, then it follows that a "high potential" magnesium anode (delivering approximately 0.3 volts more than regular magnesium) can give you an even lower overall cost. This would be especially true if there is no sacrifice in useful ampere hours of current per pound.

Galvomag, the high-potential magnesium anode developed by The Dow Chemical Company, meets both of these requirements: It delivers 20 to 35 per cent more current than standard H-1 alloy magnesium anodes; and it has a current efficiency (ampere hour rating) comparable to H-1 anodes. Other so-called "high potential" and "high current" anodes tested, however, do not show current efficiencies in a comparable range.

To help you get the most for your magnesium anode dollar, we suggest that you specify Galvomag or its equivalent in solution potential and ampere hour rating.

CSI engineers—pioneers in cathodic protection—have also developed a wide variety of special sizes and shapes to make Galvomag even more efficient. For example, the use of these special sizes in "hot spot" protection of pipe lines makes it possible to lower the number of anodes required by one-third—without decreasing the amount of protection or shortening the life of the installation.

It will pay you to check with CSI—for expert engineering and installation services, plus a complete line of cathodic protection supplies for both rectifier and anode installations. Prices are competitive. Call or write today.

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NATIONAL and REGIONAL MEETINGS and SHORT COURSES

1962

January 17-19—Canadian Region Eastern Division Conference, King Edward Hotel, Toronto, Ontario.

February 7-9—Canadian Region Western Division Conference, Calgary, Alberta.

March 18—NACE Board of Directors Meeting, Muehlebach Hotel, Kansas City, Mo.

March 19-23—18th Annual Conference and 1962 Corrosion Show, Kansas City, Missouri, Municipal Auditorium.

March 23—NACE Board of Directors Meeting, Muehlebach Hotel, Kansas City, Mo.

October 1-4—Northeast Region Conference, Hotel Sheraton Ten Eyck, Albany, N. Y.

October 9-11—North Central Region Conference, Detroit, Mich.

October 11-12—Southeast Region Conference, Birmingham, Ala.

October 16-19—South Central Region Conference and Exhibition, Granada Hotel, San Antonio.

October—Western Region Conference, San Diego, Cal.

1963

March 18-22—19th Annual Conference and 1963 Corrosion Show, Convention Hall, Atlantic City, N. J.

October 14-17—South Central Region Conference, Oklahoma City, Okla.

October 22-24—Northeast Region Conference, Niagara Falls, N. Y.

1964

March 9-13—20th Annual Conference and 1964 Corrosion Show, Sherman Hotel, Chicago, Ill.

October 19-22—South Central Region, Statler-Hilton Hotel, Dallas, Texas.

October 25-30—North Central Region Conference, Hotel Lemington, Minneapolis, Minn.

1965

March 15-20—21st Annual Conference and 1965 Corrosion Show, Chase-Park Plaza Hotel, St. Louis, Mo.

SHORT COURSES

1962

February 1-2—Houston Section Corrosion Control Short Course, Rice Hotel, Houston, Texas.

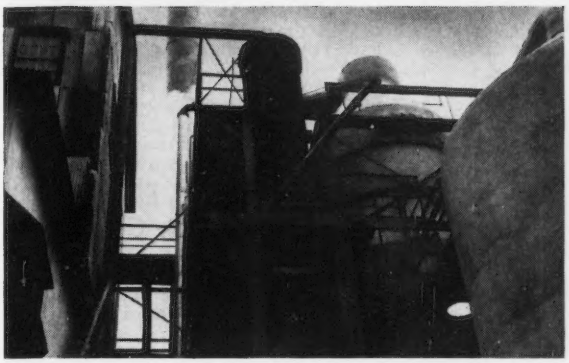
Feb. 21-23—12th Annual Corrosion Short Course, Mayo Hotel, Tulsa, Okla.

June 13-15—7th Annual Appalachian Underground Corrosion Short Course, University of West Virginia, Morgantown, W. Va.

Sept. 26-28—9th Annual Central Oklahoma and University of Oklahoma Corrosion Control Short Course, Norman.

New refining process at International Salt shows...

How MONEL Alloy 400 checks corrosion by NaCl



Step 1: Rock salt slurry is heated by direct contact with vapor from first-effect evaporator, dissolving half the salt. Internal heater parts are of MONEL alloy 400. Steam is injected from second heater, bringing temperature to 222°F. Hot brine exits to saturator and thickener where salt is completely dissolved and Ca_2SO_4 separated.

At their Avery Island, La. mine, The International Salt Company has the first full-scale salt refinery based on the Recrystallizer process. This revolutionary method of salt refining utilizes the difference in solubility of NaCl and its major impurity Ca_2SO_4 to produce 99.99% pure salt.

The economical Recrystallizer process:

—**eliminates** costly lime and soda ash treatments used in conventional salt refining.

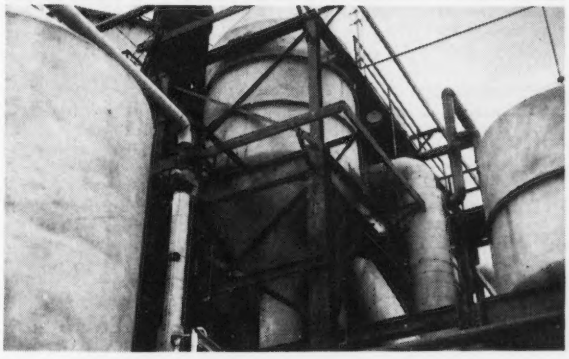
—**eliminates** tubular heat exchangers, reducing maintenance.

—**eliminates** two of the four expensive evaporators used in conventional refineries. Employs a double-effect Recrystallizer instead of a quadruple-effect forced-circulation unit.

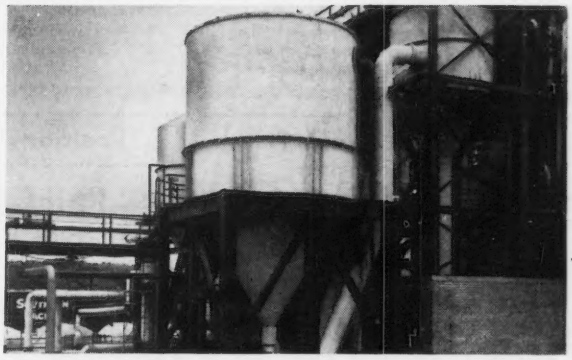
—**minimizes** the chance of salt contamination through the use of MONEL* alloy 400 in the settlers, evaporators and heaters. The outstanding resistance of this nickel-copper alloy to chloride corrosion protects product purity.

For more information about MONEL alloy 400, write for technical bulletin T-5.

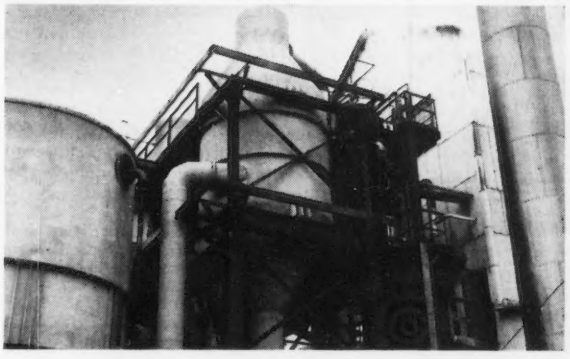
*Registered trademark



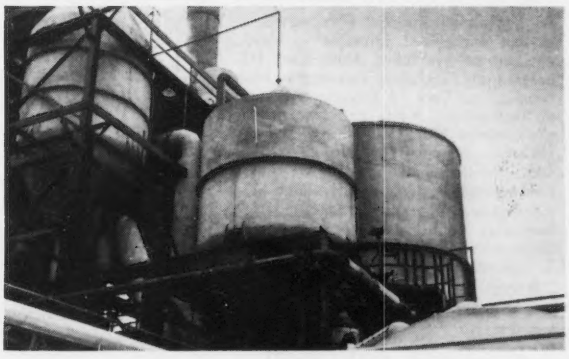
Step 2: Hot brine is completely clarified at 220°F and flash cooled to about 180°F, releasing vapor which is used in the first contact-heater. Concentration of brine plus reduction of temperature precipitates some pure salt crystals. First effect evaporator is made of Alloy 400.



Step 3: Recrystallized sodium chloride, suspended in saturated brine, is handled by this first effect settler—made of MONEL alloy 400. The slurry is thickened so that clarified mother liquor can be pumped to the second effect settler for further evaporation. Approximate temperature is 175°F.



Step 4: Hot brine at 175°F enters the second effect flasher from the first effect settler and is flash cooled to 130°F. This yields salt and vapor. Crystallized salt settles down elutriating legs and is withdrawn periodically. Temperature range is 120-140°F. Metal for the unit is MONEL alloy 400.



Step 5: Slurry from the second effect flasher is thickened in the second effect settler. The clarified brine is then pumped back to the feed tank for slurrying more rock salt. The second effect settler is made of steel clad with MONEL alloy 400. This plant produces 15 tons of 99.99% pure salt every hour, around the clock.

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Southeast Region

Birmingham Elects Officers for 1962

J. C. Ray of Southern Bell Telephone & Telegraph Co., has been elected the 1962 chairman of the Birmingham Section.

Other new officers are D. D. Holmes of Alabama Power Co., vice chairman, and Ed Lindley of Southern Bell Telephone & Telegraph Co., assistant secretary-treasurer. These officers were elected at the fourth quarter meeting of the section at the Molton Hotel on November 3.

Marion M. Fink, present chairman, reported that when the Junior Engineers' Training Program sponsored by the Engineering Council of Birmingham and Jefferson County has been worked up, it is expected that NACE will be requested to furnish someone to teach corrosion.

Guest speaker Ralph M. Hurst of Mississippi Valley Gas Company, Jackson, told of his company's cathodic protection practices, started in 1941, that have reduced gas losses from 4.16% of total purchased to 3.08%. At present the gas company has 2434 miles of 3-inch pipe cathodically protected. His talk, which included improvements made in installing deep well ground beds, was followed by questions from the floor.

Edward E. Gross Elected Tidewater's New Chairman

The Tidewater Section has elected officers for 1962. They are Chairman Edward E. Gross of Norfolk Redevelopment & Housing Authority, Norfolk, Va., Vice Chairman William D. Lyliston, Jr., of Newport News Shipbuilding & Dry Dock Company, Newport News, Va., and Secretary-Treasurer Daniel C. Wimer of Commonwealth Natural Gas Corporation, Richmond, Va.

POSITIONS WANTED and AVAILABLE

Active and Junior NACE members and companies seeking salaried employees may run two consecutive advertisements annually without charge under this heading, not over 35 words set in 8 point type. Advertisements to other specifications will be charged for at $12.50 a column inch.

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MANUFACTURERS' REPRESENTATIVES
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With background in corrosion prevention materials to intelligently present protective coating systems exclusively designed for architectural, maintenance and technological use. Conventional paint salesmen will not qualify for these openings. Extensive technical assistance available, sustained by more than quarter century exclusively in specialized coating field. Coatings involve wide range of custom engineered formulae in vinyls, epoxies, polyurethanes, Neoprenes, Hypalons*, etc., including specification materials; i.e., Corp of Engineers, Bureau of Reclamation, A.E.C. and special missile coatings. More than 70 qualified applicators presently established nation wide. Territories protected on an exclusive contract basis. Special training provided. Local interviews. For further information write directly to Surface Engineering Co., Inc., Box 2273, Wichita 1, Kansas.

* Registered trade mark of Du Pont

Positions Available

Wanted: Chemical or metallurgical engineer for laboratory work on corrosion of copper and aluminum and their alloys. Location: New York State. CORROSION, Box 61-30.

Wanted: Engineer with corrosion experience to head up department dealing with coatings, sand blasting, external pipe coats, and cathodic protection in South Louisiana. CORROSION, Box 61-29.

Positions Available

Chief Chemist
RESEARCH & DEVELOPMENT
PAINTS

Medium size Eastern manufacturer making well-established line of chemical-resistant coatings has challenging opportunity for experienced chemist in development and formulation of coatings based on chlorinated rubber, vinyls, alkyds, epoxies, acrylics, coal tar pitch, asphalt.

Duties also include trouble-shooting requiring broad paint experience. Present chief chemist approaching retirement. Remuneration commensurate with ability. Replies will be kept strictly confidential. Send complete resume preferably accompanied by photo, and indicate salary requirements. Address CORROSION, Box 61-27.

Positions Wanted

Consulting Chemist with worldwide reputation on paints, lacquers and allied coatings available for specifications, sales service, trouble shooting, literature research and formulation. Expert on corrosion problems. Creative, sales-minded, experienced writer and speaker. CORROSION, Box 61—26

SALES MANAGER

Age 35, 14 years' sales experience, 8 years' successful experience in all phases of water treatment and protective coatings including 2 years as sales manager of 10 salesmen. Top references, New England preferred. CORROSION, Box 61-28.

SECTION CALENDAR

December

4 Central Oklahoma Section
6 Teche Section
7 West Kansas Section
7 Pittsburgh Section, Underground Corrosion Round Table, Chairman C. A. Erickson of Peoples Natural Gas Company
8 Rocky Mountain Section
12 Twin Cities Section
12 Houston Section, Chemical Industry Development in Texas and Educational Trends in Engineering Schools by J. D. Lindsay of Texas A&M College. Ladies Night.
13 Greater Boston Section, Contributions of Electrochemistry to Corrosion Control by Herbert H. Uhlig of Massachusetts Institute of Technology.
19 Alamo Section
19 Philadelphia Section, Management's Concern With Corrosion by G. E. Best

January

2 Shreveport Section, Economics of Coating for Corrosion and Paraffin Prevention in Oil Well Tubing by Barnard P. Goodman of Tube-Kote, Inc.
4 Pittsburgh Section, Corrosion of Steel in Nuclear Power Reactors by A. L. Medin of U. S. Steel Corp.
9 Twin Cities Section
9 Houston Section, Corrosion Problems in Production Equipment by F. A. Prange of Phillips Petroleum Co.
16 Chicago Section, Corrosion Inhibitors—Theory and Application by J. I. Bergman of Armour Research Foundation.
18 Kanawha Valley Section, Charleston, W. Va., Chemical.
30 Schenectady-Albany-Troy Section, Manufacturing Controls to Establish Corrosion Resistance of Zirconium Alloys by R. J. Enrico of Knolls Atomic Power Laboratory.

19 Symposia Set For National Meet In Kansas City, Mo.

Nineteen symposia are scheduled for presentation at the 18th Annual NACE Conference in Kansas City, Missouri, March 19-23. Among these will be recent advances in theoretical corrosion. This symposium, the first of the conference, starts at 9 am, March 19. It promises to be one of the highlights of the national meeting. Eight distinguished corrosion researchers from highly esteemed industrial, government and university laboratories will present papers (see related story on Pages 60-61).

Headquarters will be Hotel Muehlebach where the first day's technical meetings will be held. Thereafter, meetings will be held in the Municipal Auditorium's 4th, 5th and 6th floor conference rooms and in the Little Theatre on the ground floor.

Corrosion Show

The downtown Municipal Auditorium, one block from Hotel Muehlebach, also will be the site of the 1962 Corrosion Show which runs March 20-22. Fifty firms to date are planning exhibits of their corrosion control products. To be staged in Exhibition Hall, the show should prove very enlightening. On display will be both new and time tested products to help you solve a great many corrosion problems. Representatives of the exhibitors will be present to explain the products and their applications and to furnish technical literature.

Also scheduled during the conference are the annual business meeting of the association, the installation of the new officers and three directors and the presentation of the Whitney and Speller Awards.

Symposia Scheduled

Symposia scheduled are cathodic protection, chemical industries, corrosion in the missile industry, corrosion inhibitors, corrosion principles, elevated temperatures, general corrosion, high purity water, marine, oil and gas production, pipe line—general, plastics, protective coatings, refining industry, utility industries, pipe line and underground corrosion—round table, general corrosion problems—round table, theoretical corrosion and aluminum pipeline.

Officers

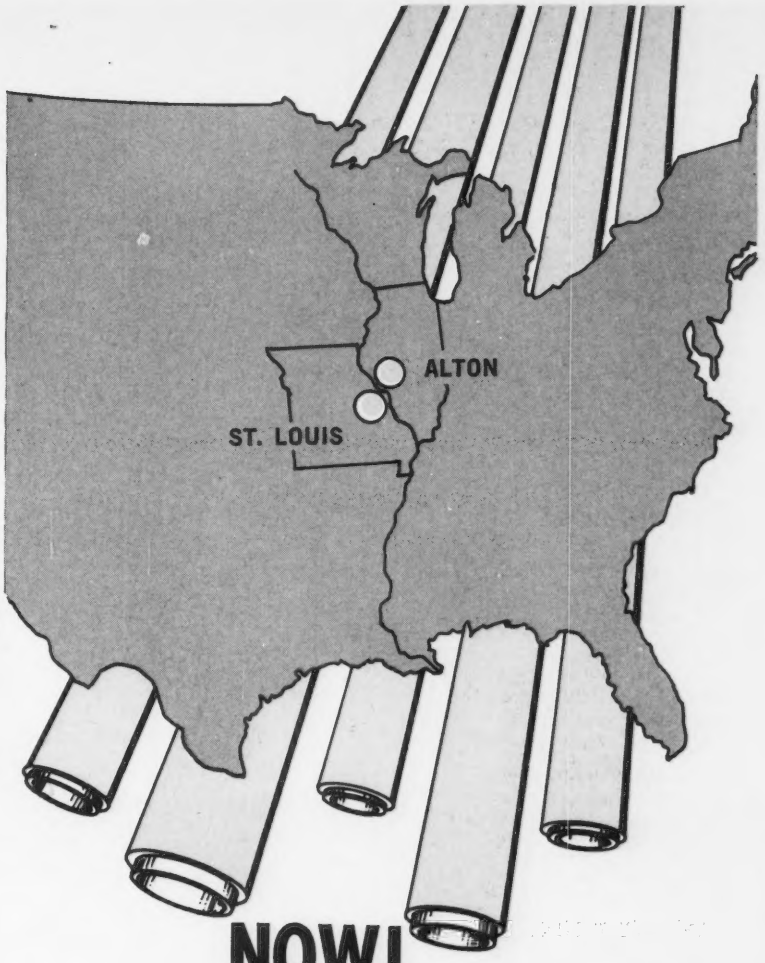
Chairman of the technical program is L. W. Gleekman of Wyandotte Chemicals Corporation, Wyandotte, Mich. His co-chairman is David Roller of Magna Products, Inc., Anaheim, Cal.

General conference chairman is Robert H. Goodnight of Cook Paint & Varnish Company, North Kansas City, Mo. Conference co-chairman is Jack L. Grady of the J. L. Grady Company, Kansas City, Mo.

Other committee chairmen are O. E. Moore of Panhandle Eastern Pipe Line Co., entertainment and fellowship; Fred H. Rogers of Great Lakes Pipe Line Co., publicity; Forrest E. Davidson of Southwestern Bell Telephone Co., properties, and John E. Wright of Gas Service Co., local arrangements.

Mrs. Robert H. Goodnight and Mrs. Jack Grady are in charge of the ladies' program.

The complete technical program will be published in CORROSION in the near future.



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from SPI's new Alton, Illinois Plant comes STANDARD X-TRU-COAT

Standard Pipeprotection's new plant at Alton, Illinois has been shipping Standard X-Tru-Coat pipe to the Southwest and West since early July. Sizes available include ¾" Nom. through 4½" O.D.

X-Tru-Coat high density polyethylene plastic coating for pipe is produced under the DeKoron process — Pat. Nos. 2,447,420 and 2,467,642 — and is extruded on pipe over a pressure sensitive adhesive.

Standard Procedures assuring top quality prevail at the Alton, Illinois plant, as they have for many years at SPI's coating and wrapping plant in St. Louis. In-Transit privileges are available at both plants from any pipe mill.

Standard Pipeprotection, with the introduction of X-Tru-Coat, has continued to contribute to the progress of the oil and gas industry.

Write for complete information and for Price List Number X-1.

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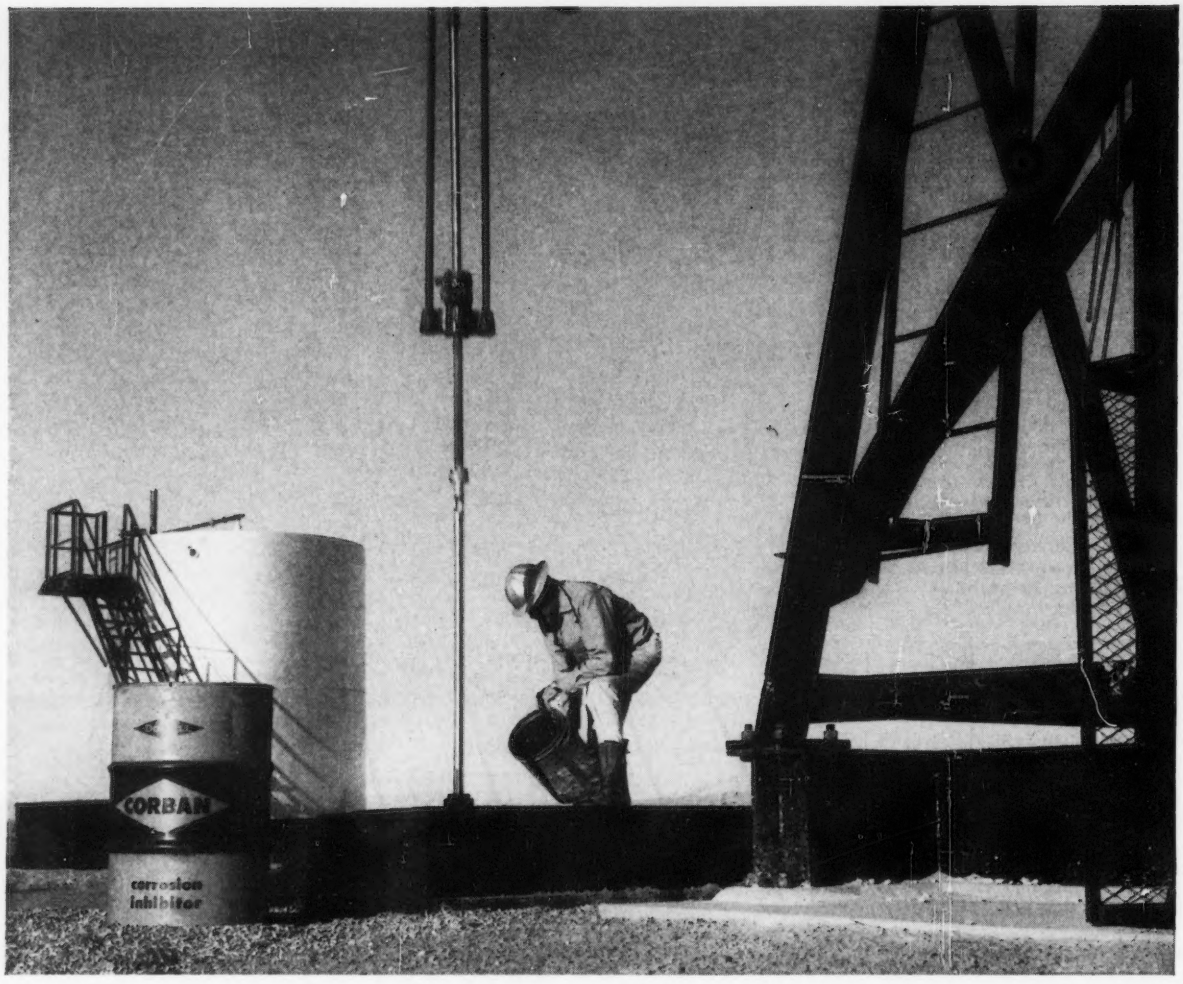

three ways Corban helps cut secondary recovery costs

Many producers are cutting expenses of secondary recovery projects by using Corban®, Dowell's family of versatile, low-cost corrosion inhibitors. Corban helps reduce costs in three important ways:

FIRST, Corban greatly extends the life of tubular goods by retarding corrosion. This holds true for surface lines as well as downhole tubing.

SECOND, Corban works two ways to reduce formation plugging. It reduces the formation of insoluble iron salts—a result of corrosion—that is a serious cause of formation plugging. It also acts as a detergent to help disperse organic deposits. In most fields, Corban helps increase water injection rates and/or decrease injection pressures.

THIRD, Corban reduces sucker rod failures caused by corrosion. Sucker rod life has been lengthened dramatically when treatments using Corban were begun.

Corban also helps control corrosion in gas wells, flowing oil wells and salt water disposal projects. Twelve different formulations are available to meet a wide range of well conditions. Ask your Dowell representative for full information. Dowell services and products are offered in North and South America, Europe, North Africa and Iran. Dowell, Tulsa 14, Oklahoma.

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Fifty Firms Contract for Booths At Kansas City Corrosion Show

Fifty firms have contracted to exhibit products at the 1962 Corrosion Show in Kansas City, Missouri, March 20-22. Being held concurrently with the 18th Annual NACE Conference, which runs March 19-23, the show promises to be an impressive array of new and tested controls. On display will be products to solve a great many corrosion problems and on hand will be representatives of the companies to give complete technical data on the products and to discuss their applications with you.

New Companies

In November CORROSION, Page 59, are listed 43 firms holding space at that time and their products. The seven additional exhibitors are A. M. Byers Company, E. I. du Pont de Nemours & Co., Inc., B. F. Goodrich Chemical Company, Minnesota Mining & Manufacturing Company, The Philip Carey Manufacturing Company, Steelcote Manufacturing Company and St. Louis Metallizing Company.

Products

Products these companies have scheduled to exhibit include wrought iron pipe, plate and bars, steel pipe, plastic pipe, pipe jackets, felts and padding, sheeting and sheet lining and protective coatings and application equipment.

Slide shows of the exhibits will be made and distributed, upon request, to NACE sections for presentation at technical meetings.

Exhibit Information

Companies desiring information concerning available space rates, and other exhibition information should contact R. W. Huff, Jr., NACE Exhibits Manager, 1061 M & M Building, Houston 2, Texas.

Bo Bardsley Succumbs

Bo Bardsley, co-owner of Remco Pipeline Equipment & Supplies, Tulsa died November 14. An early member of NACE, he held membership No. 643, dated April 10, 1946. Mr. Bardsley, for many years connected with companies serving the pipeline field, was employed by Crutcher-Rolfs-Cummings and Jacobs Electric Company before forming his own company late in 1953.

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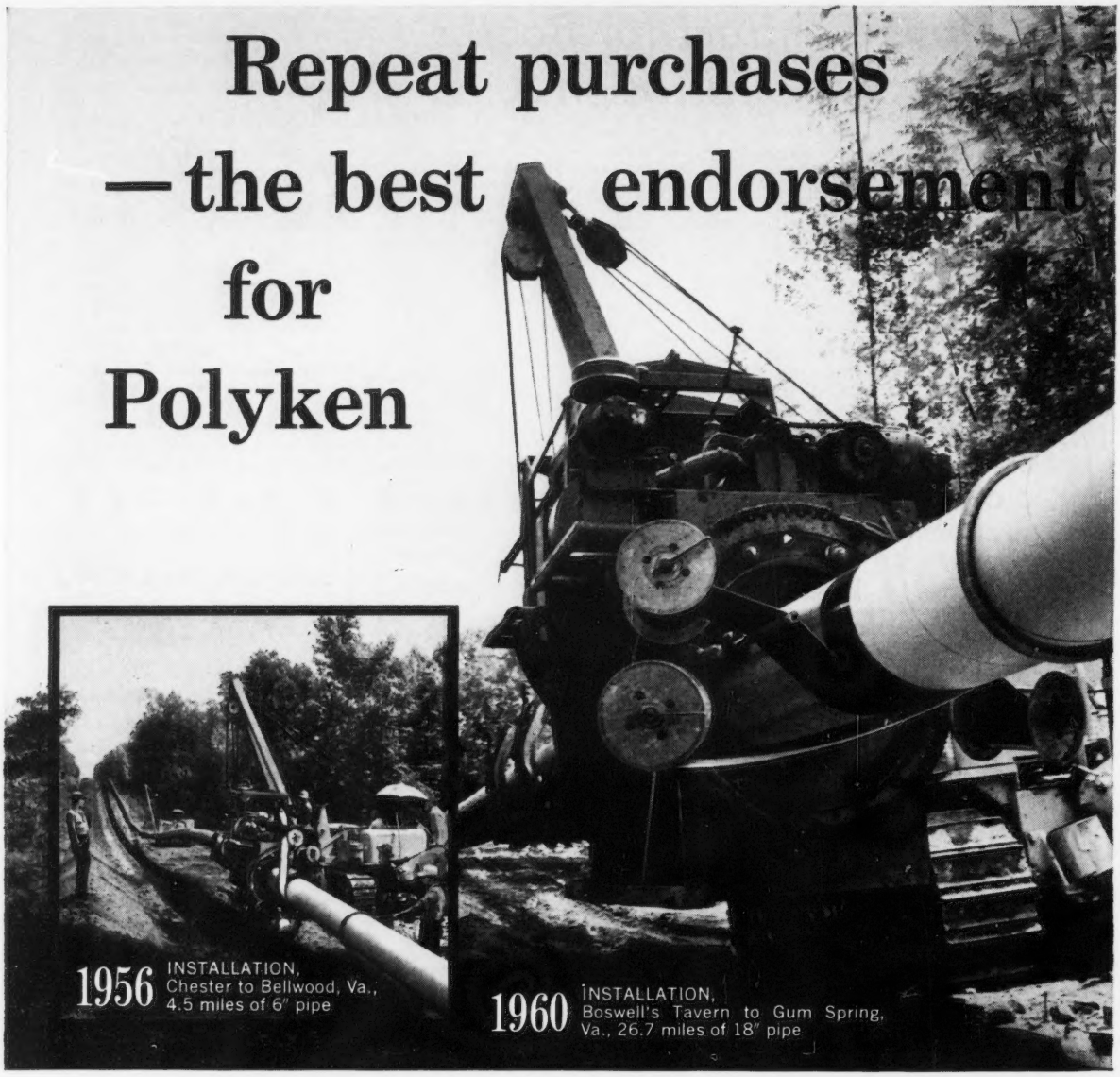

1956 INSTALLATION, Chester to Bellwood, Va., 4.5 miles of 6″ pipe

1960 INSTALLATION, Boswell's Tavern to Gum Spring, Va., 26.7 miles of 18″ pipe

Commonwealth Natural Gas makes repeated use of Polyken® tape on strength of past performance

Officials of Commonwealth Natural Gas Corporation of Richmond, Va., were impressed in 1956 with the many advantages of Polyken protection. With confidence based on the cathodic protection needs of their Polyken tape-coated 1956 Bellwood line (*inset photo*), they put this coating to work two years later on their Chester to Petersburg, Va., line (12 miles of 18″ pipe). In 1959, *more* Polyken coating . . . Waverly to Ivor, Va., (15.8 miles of 12″ pipe). And most recently, the line from Boswell's Tavern to Gum Spring (*main illus.*).

It stands to reason that repeated use is founded on first-hand satisfaction and trust. Many discriminating companies make Polyken protection their standard specification because of its dependable performance. Get full details on Polyken performance and economy from your representative, or write Polyken Sales Division, 309 West Jackson Boulevard, Chicago 6, Illinois.

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Plainfield, New Jersey
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Draley and Larrabee Selected To Receive NACE's Top Awards

Two eminent corrosion experts have been selected to receive NACE's 1961 Whitney and Speller Awards. These awards, the highest bestowed by NACE, will be presented at the banquet during the 1962 NACE Annual Conference in Kansas City, Mo., March 19-23.

J. E. Draley of Argonne National Laboratory, Lemont, Ill., will receive the Willis Rodney Whitney Award in recognition of outstanding public contributions to corrosion science. C. P. Larrabee of the Applied Research Laboratory, United States Steel Corporation, Monroeville, Pa., will receive the Frank Newman Speller Award in recognition of outstanding public contributions to corrosion engineering.

The two awards, named for the original recipients, were first presented in 1946. G. E. Best, immediate past president of NACE and chairman of the Awards Committee, will make the presentations.

J. E. Draley

Mr. Draley has been engaged in corrosion research since 1942 when he joined the Metallurgical Laboratory, University of Chicago. More recently he has been associated with Kellex Corporation, Oak Ridge National Laboratory and Argonne National Laboratory working on atomic energy projects. His work has been largely with corrosion mechanisms of aluminum, uranium and zirconium and their alloys in pure water and dilute solutions.

He has written numerous "projects" publications and has been a frequent speaker on corrosion subjects for NACE, the Electrochemical Society and other technical groups.

He has served as chairman of the Corrosion Division of the Electrochemical Society, as Corrosion Division editor for the Journal of the Electrochemical Society, as chairman of the Gordon Research Conference on Corrosion and as a member of the committee for fundamental work on corrosion for the Corrosion Research Council. Also, he was instrumental in setting up annual symposia on corrosion under the auspices of the Atomic Energy Commission. He initiated an international meeting on aqueous corrosion of reactor materials in Brussels in 1959 under the joint sponsorship of EURATOM and the U. S. Atomic Energy Commission.

He is currently a member of the Corrosion Resistant Metals Committee of the Institute of Metals Division of the Metallurgical Society of the American Institute of Mining and Metallurgical Engineers. He is also a member of a special subcommittee on light metals for nuclear applications of the American Society for Testing and Materials.

C. P. Larrabee

Mr. Larrabee, chief research engineer—corrosion prevention at U. S. Steel Corporation's Applied Research Laboratory in Monroeville, Pa., joined NACE in 1944. Since 1935 he has been concerned primarily with research work on corrosion of steel products, particularly in the area of atmospheric and other natural media exposures.

He is the author of numerous technical papers and a frequent lecturer on various phases of corrosion control. On November 27 of this year he presented his latest paper entitled Corrosion of Steel As Influenced by Its Composition and Environment at the Southeast Region Conference in Miami.

He has served as a director of NACE and as chairman of the Technical Prac-

(Continued on Page 59)

J. E. Draley
Whitney Award

C. P. Larrabee
Speller Award

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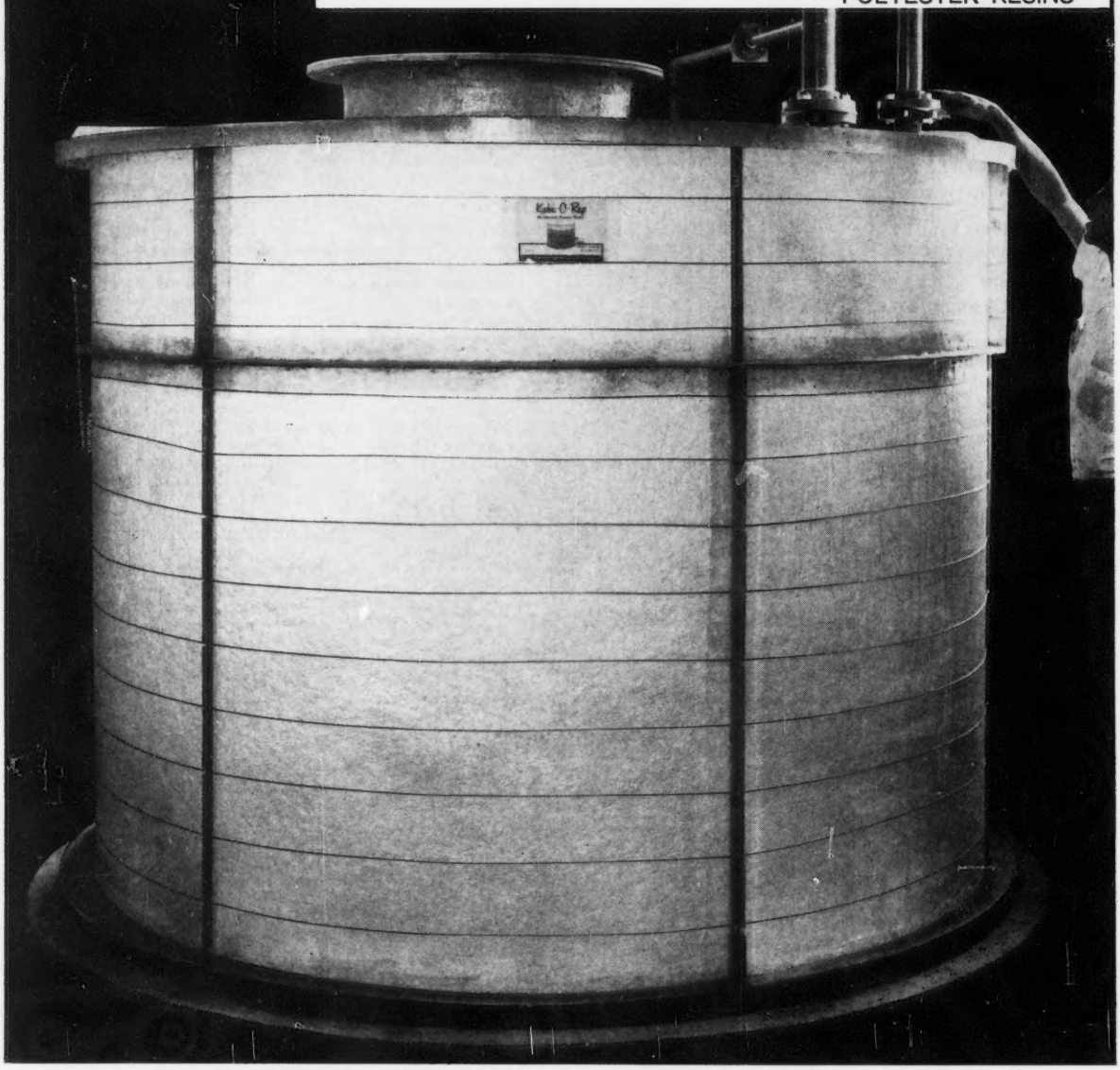

Problem: Get a 7′ tank through a 3′ door

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What's more, North Tonawanda's large Kabe-O-Rap tank, as well as their 3′ version, will fight off the acid attack of the city's 23% hydrofluosilicic acid indefinitely. They need no painting or other costly maintenance. Size and location of outlets can be changed. If repairs become necessary they can easily be made by patching with Laminac and fibrous glass.

Learn more about Laminac, the material that has been solving industry's toughest corrosion problems. For details or technical assistance, contact any Cyanamid office listed below.

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CYANAMID

Whitney and Speller Awards

(Continued From Page 57)

tices Committee. He is an active member of Technical Committees T-4D, T-5E, T-6G and T-6R. In addition to his many NACE activities, he is also active in committee work of other scientific and technical societies. Currently he is chairman of the Advisory Committee on Corrosion of the American Society for Testing and Materials.

Mr. Larrabee holds a B.S. in chemical engineering from the University of Maine at Orono. For ten years prior to his association with the Applied Research Laboratory, he was a member of the staff of the Chemistry Division of the National Bureau of Standards.

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Corrosion Behavior of Zirconium-Uranium Alloys in High Temperature Water, by W. E. Berry and R. S. Peoples.

Corrosion and Water Purity Control for the Army Package Power Reactor by R. J. Clark and A. Louis Medin.

Removal of Corrosion Products from High Temperature, High Purity Water Systems with an Axial Bed Filter by R. E. Larson and S. L. Williams.

Some Relations Between Deposition and Corrosion Contamination in Low Make-up Systems for Steam Power Plants by E. S. Johnson and H. Kehmna.

Remittances must accompany all orders for literature the aggregate cost of which is less than $5. Orders of value greater than $5 will be invoiced if requested. Send orders to National Association of Corrosion Engineers, 1061 M & M Bldg., Houston, Texas. Add 75c per package to the prices given above for Book Post Registry to all addresses outside the United States, Canada and Mexico.

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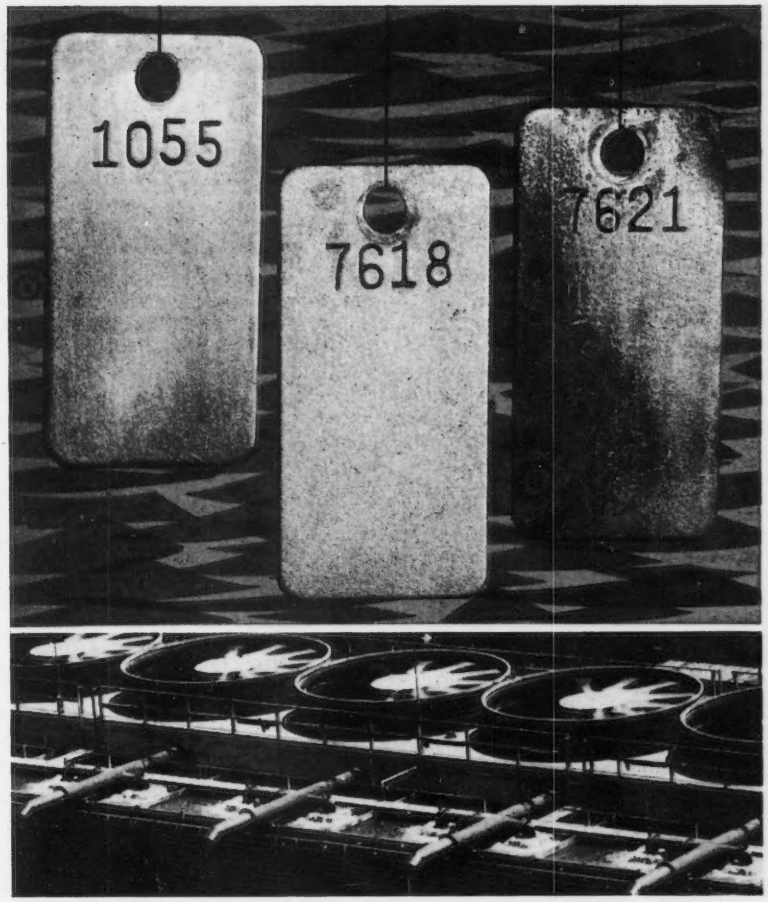

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Plant tests designed to determine which corrosion inhibitor provides the *lowest cost per degree of protection* usually indicate Hagatreat as the winner by a wide margin. Here are the results on test strips from one such test:

			corrosion rate	
Specimen No.	Location	Treatment	mg/dm²/day	mils/yr
1055 (copper)	Blowdown	Hagatreat 168	0.41	0.06
7618 (steel)	Tower Sump	Hagatreat 168	0.58	0.11
7621 (steel)	Blowdown	Hagatreat 168	1.1	0.20

The low corrosion rate registered here is typical of Hagatreat results in plants all over the country. Hagatreat is effective with steel, copper, aluminum; in fact, complete treatment as recommended by the Hagan engineer is designed to protect the entire system, from cooling towers on through.

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Special Theoretical Corrosion

Eight Outstanding Research Authorities To Be Speakers

Eight of this country's most outstanding research authorities in corrosion will be speakers at the special symposium on Recent Advances in Theoretical Corrosion which will be part of the technical program of the 18th Annual NACE Conference. This national conference to be held March 19-23 in Kansas City, Mo., will have an estimated attendance over 2000, representing persons from a wide variety of industries in this country and several foreign nations.

The eight speakers represent some of the leading laboratories concerned with corrosion research—from outstanding industrial, government and university laboratories. Their published technical articles and books include some of the most significant work in corrosion science and engineering.

This symposium on recent advances in theoretical corrosion will include discussions on fundamental corrosion research, electrochemical techniques for corrosion studies, advances in understanding organic and inorganic inhibitors, and the influence of metallurgical factors on corrosion. Other topics to be included are modern methods of alloy design and study and modern concepts of stress corrosion cracking.

The theoretical corrosion symposium will be one of the 19 symposia which will make up the technical program for the Kansas City meeting. The symposia will cover most aspects of corrosion control problems confronting industry today, ranging from missiles to the petrochemical industries.

Symposium Schedule

The theoretical corrosion symposium will begin at 9 am on March 19, the first day of the NACE Conference, with an introduction to the symposium by its chairman, Milton Stern, Manager of Research for the Linde Company's Speedway Laboratories, Indianapolis, Ind.

Schedule of topics and the speakers for the full day symposium are given below:

9:10-10:00 Contributions of Fundamental Corrosion Research, by H. H. Uhlig of Massachusetts Institute of Technology, Cambridge, Mass.

10:00-10:50 Recent Advances in Electrochemical Techniques for Corrosion Studies, by A. C. Makrides of Socony Mobil Oil Co., Inc., Dallas, Texas.

10:50-11:10 Intermission

11:10-12:00 Recent Advances in Understanding of Inorganic Inhibitors, by G. H. Cartledge of Oak Ridge National Laboratory, Oak Ridge, Tenn.

12:00- 1:00 Lunch

1:00- 1:50 Recent Advances in Understanding of Organic Inhibitors, by Norman Hackerman of the University of Texas, Austin, Texas.

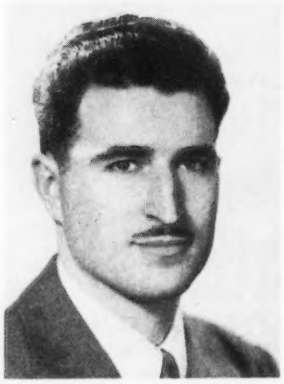
1:50- 2:40 Modern Methods of Alloy Design and Study, by N. D. Greene of Rensselaer Polytechnic Institute, Troy, N.Y.

2:40- 3:00 Intermission

3:00- 3:50 Influence of Metallurgical Factors on Corrosion, by M. A. Streicher of E. I. du Pont de Nemours & Co., Wilmington, Del.

3:50- 4:40 Modern Concepts of Stress Corrosion Cracking, by Sidney Barnartt of Westinghouse Electric Corp., Pittsburgh, Pa.

4:40- 5:00 General Discussion.

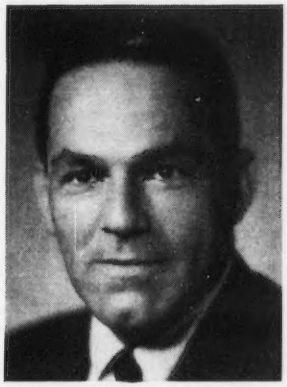


SIDNEY BARNARTT is manager of the Electrochemistry Section of Westinghouse Electric Corporation's Research Laboratories at Pittsburgh, Pa. He has a PhD in physical chemistry from the University of Toronto. He has published technical articles on corrosion, electrode kinetics, electrodeposition, potential mapping, and ultrasonic effects in electrochemistry. He is vice chairman of the Theoretical Electrochemistry Division of the Electrochemical Society and is a member of the Faraday Society and American Chemical Society.

G. H. CARTLEDGE has been a Group Leader in the Chemistry Division of the Oak Ridge National Laboratory, Oak Ridge, Tenn., until his retirement in March, 1961, but he continues his research activities as a consultant for the division. His recent research activities have been in the field of corrosion inhibition and electrode kinetics. Some of his work included development of the use of technetium in the form of the pertechnetate ion as an efficient corrosion inhibitor for iron and steel. He is a graduate of Davidson College in North Carolina and the University of Chicago. After receiving his PhD, he spent 40 years in academic positions, chiefly at Johns Hopkins University and the University of Buffalo before joining the Oak Ridge Laboratory in 1951.

NORBERT D. GREENE is assistant professor and director of the Corrosion Research Laboratory in the Department of Materials Engineering at Rensselaer Polytechnic Institute, Troy, N.Y. Before joining the faculty at Rensselaer, he was employed by the Metals Research Laboratories of Union Carbide Metals Co., Niagara Falls, N.Y. He has a BS from the University of Rochester and an MS and PhD in metallurgical engineering from Ohio State University. He received the NACE's 1959 Young Author Award and is active in NACE as vice chairman of the Editorial Review Sub-Committee. He also is a member of the American Society for Metals, the Electrochemical Society and the Electrochemical Society of Japan. He has published several technical articles on pitting corrosion, passivity and various corrosion problems related to stainless steels.

Symposium Set for Conference



NORMAN HACKERMAN is vice president and provost of the University of Texas. Formerly, he was professor and chairman of the Chemistry Department and director of the Corrosion Research Laboratory. He also was dean of the university's Research and Sponsored Programs. His research has dealt primarily with the chemistry and physics of solid surfaces. During the past few years, he and his students have published several articles on adsorption on metal surfaces and its influence on electrochemical properties and reactivity, on the passivity of metals and on energetics of solid-liquid interfaces. He has a PhD in physical chemistry from Johns Hopkins University. An active member of NACE, he has served on the association's Board of Directors and is chairman of the NACE Committee on Education. He received the NACE 1956 Whitney Award, one of the association's two top awards for outstanding work in the science and engineering of corrosion. He also is a member of the American Chemical Society, Electrochemical Society, American Association for the Advancement of Science, the Faraday Society and is a Fellow of the New York Academy of Sciences.

MILTON STERN is manager of research at the Speedway Laboratories of the Linde Company, a division of Union Carbide Corporation, Indianapolis, Ind. An active member of NACE, he is past chairman of the association's Niagara Frontier Section. He has a BS in chemical engineering from Northeastern University and an SM and ScD from Massachusetts Institute of Technology in physical metallurgy. He was Weirton Postdoctoral Fellow at the Corrosion Laboratory of MIT for two years before joining Union Carbide. He received the Electrochemical Society's Young Authors Prize in 1955 and 1958 and is the society's Corrosion Division Editor. His published work has been in the areas of corrosion, electrochemistry, reaction kinetics and physical metallurgy.

H. H. UHLIG is professor of metallurgy at Massachusetts Institute of Technology and is in charge of the Corrosion Laboratory. His undergraduate degree in chemistry was from Brown University and his PhD from MIT. He has contributed numerous papers to the scientific and engineering literature in the fields of corrosion, electrochemistry and metallurgy. He edited the "Corrosion Handbook" published in 1948. He received the NACE Willis R. Whitney Award for his achievements in the science of corrosion. He was president of the Electrochemical Society in 1955-56 and helped found the Corrosion Division of that society. He was Guggenheim Fellow at the Max Planck Institute fur Physikalische Chemie in Gottingen in 1961 and was plenary lecturer at the First International Congress on Metallic Corrosion held April, 1961, in London. He also has received the 1961 Palladium Medal of the Electrochemical Society.

A. C. MAKRIDES is associated with the Field Research Laboratory of Socony Mobil Oil Company, Dallas, Texas. His research has been concerned with dissolution of metals, hydrogen overpotential and corrosion inhibition. He also has done work on the heat of wetting of solids and on the surface properties of silicon dioxide. He has a PhD from the University of Texas and was formerly affiliated with the Metals Research Laboratories of Union Carbide Metals Company. During 1954-55, he was a Union Carbide Research Fellow at the Institute for the Study of Metals at the University of Chicago and from 1955 to 1957 was a research chemist with the American Petroleum Institute at the University of Texas Department of Chemistry. He is a member of the Electrochemical Society, the Faraday Society, and the American Chemical Society. In 1957, he received the Electrochemical Society's Turner Memorial Award and the 1960 Young Author's Prize.

MICHAEL A. STREICHER is a research associate in the Materials Technology Section of the Engineering Research Laboratory at E. I. du Pont de Nemours & Company, Wilmington, Del. He has a PhD in metallurgy from Lehigh University. His publications include articles on theory of metal dissolution, passivity and pitting resistance of stainless steels, corrosion inhibition, development of evaluation tests for intergranular corrosion of stainless steels, immersion plating and conversion coatings on metals. In 1958-60 he was chairman of the Corrosion Division of the Electrochemical Society and is chairman of the 1962 Gordon Conference on Corrosion.

• A summary of the technical program for the 18th Annual NACE Conference, March 19-23, 1962, in Kansas City, Mo., will be published in the January issue of the new NACE magazine MATERIALS PROTECTION, which will be sent to all NACE members in addition to CORROSION magazine.

• Subscriptions to the new magazine MATERIALS PROTECTION for nonmembers can be obtained by writing NACE at 1061 M & M Bldg., Houston 2, Texas.

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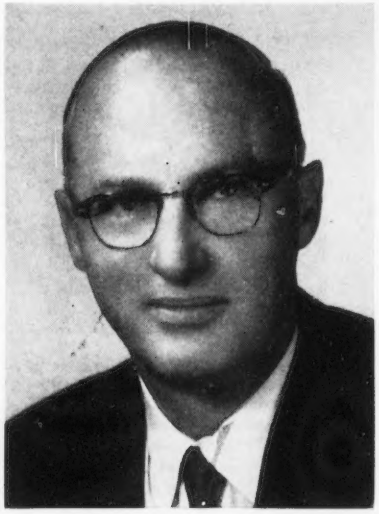
New Officers, Three Directors To Be Installed at Kansas City

Rolland McFarland, Jr., secretary and technical director of Hills-McCanna Company, Chicago, has been elected president of NACE for 1962-63 and C. G. Munger, vice president of Amercoat Corporation, South Gate, Cal., vice president. C. G. Gribble, Jr., district manager for Metal Goods Corporation, Houston, has been re-elected treasurer.

These officers will be installed next March 23 at NACE's 18th Annual Conference and 1962 Corrosion Show at Kansas City, Mo.

Also elected were three directors for three-year terms. W. H. Burton, materials consultant in charge of the materials engineering and corrosion section of the engineering department of Allied Chemical Corporation, General Chemical Division, Camden, N. J., will represent corporate members. Otto H. Fenner, corrosion specialist with Monsanto Chemical Company, St. Louis, Mo., and M. E. Holmberg, metallurgical consultant, Houston, will represent active members.

Rolland McFarland, Jr.
President



C. G. Munger
Vice President

Rolland McFarland, Jr.

President-elect McFarland, the incumbent vice president and the author of several technical papers published in CORROSION, has been a member of NACE since 1948. He has served as a director representing active members of NACE, technical program chairman for the 1959 NACE Annual Conference and chairman of Technical Committee T-6A, Organic Coatings and Linings for Resistance to Chemical Corrosion. He is a member also of the American Institute of Chemical Engineers, the American Chemical Society and the American Society for Metals. He was graduated from Armour Institute of Technology's

C. G. Gribble, Jr.
Treasurer

School of Chemical Engineering and did graduate work in organic chemistry at the University of Chicago. For more than 20 years he has been engaged in design and application of pumps, valves and allied equipment.

C. G. Munger

C. G. Munger, the newly elected vice president, has served as a director representing NACE corporate members, chairman for four years of Technical Committee T-6A, Organic Coatings and Linings for Resistance to Chemical Corrosion, and a member of the Awards Committee. He became a member of NACE in 1937.

(Continued on Page 64)

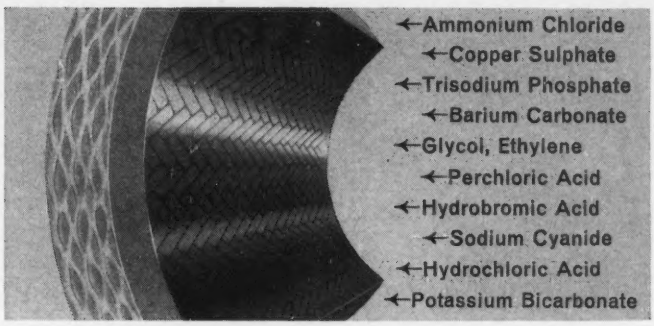
Attention Librarians

and others who bind CORROSION by Volumes!

Those who bind CORROSION by volumes and who customarily extract Technical Section pages are reminded that it will be desirable to extract also the Technical Topics Section and add it to the Technical Section of each issue. Although the Technical Topics Section does not have cumulative numbering of pages, it will be relatively easy to locate articles if this procedure is followed.

- Effective in 1962 CORROSION'S annual index will cover only the articles in CORROSION. The index will be published as a part of the December issue.

- NACE's new monthly magazine MATERIALS PROTECTION will have an annual index separate from that in CORROSION.



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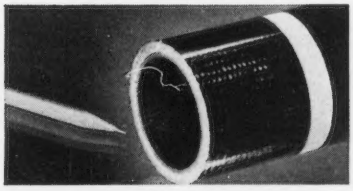

Fibercast has built-in inhibitors you can depend on to combat corrosion, contamination, scaling, electrolytic action. It is capable of withstanding sudden temperature differentials from cool liquids to hot gases or steam. It's good for all operating pressure ranges to 1200 psi.

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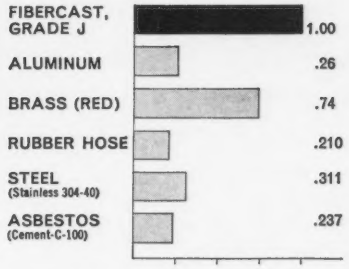

*Basing Fibercast as unit life of 1 and others as comparative percentages thereof.

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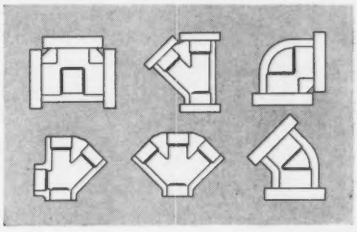

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Three New Directors Elected

W. H. Burton

O. H. Fenner

M. E. Holmberg

(Continued From Page 62)

He is the author of numerous technical articles on corrosion and has received several patents on corrosion control materials and methods. He is a member of the American Institute of Chemists, the American Chemical Society and the Paint, Lacquer and Varnish Association. After receiving a B.A. in chemistry from Pomona College in Claremont, Cal., he did graduate research work for two years on resin formation and polymerization at Claremont College.

C. G. Gribble, Jr.

Charles G. Gribble, Jr., the re-elected treasurer, has been active in NACE affairs since joining in 1948. He, a corporate member since 1954, has served as a director representing corporate members, local arrangements commitee chairman for the 1952 Annual Conference and co-chairman for exhibits at the 1957 Annual Conference. He also has been chairman of the Houston Section.

W. H. Burton

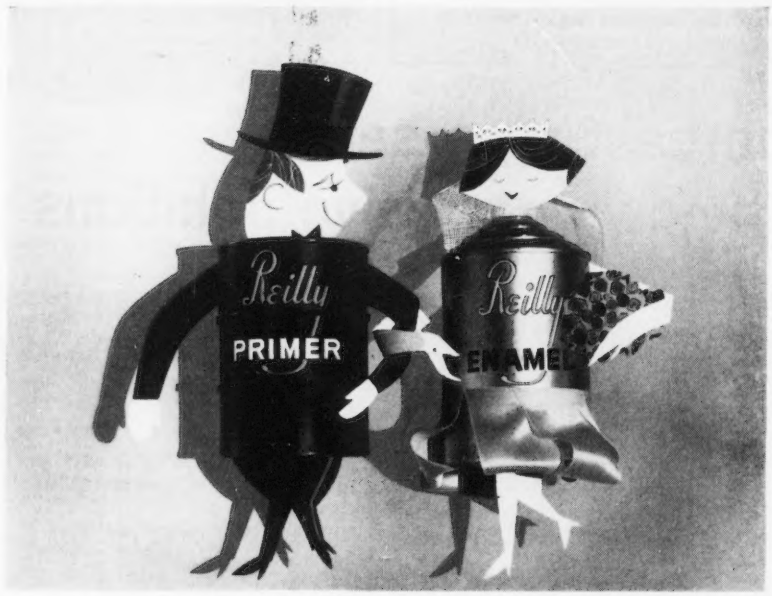
W. H. Burton, elected as a director representing corporate members, has been a member of NACE since 1953. He has been chairman of Technical Committee T-5, Corrosion Problems in the Process Industries, an officer of the Philadelphia Section and the representative of NACE on an American Standards Association sectional committee. He also is a member of the High Alloy Steel Committee of the Welding Research Council and the Atomic Energy Commission Welding Forum and is vice chairman of the American Society for Testing and Materials' Committee C-3 on Acid Resistant Mortars. He is a registered professional engineer, holding an M.S. in industrial chemistry and an M.E. in mechanical engineering from Stevens Institute of Technology.

Otto H. Fenner

Otto H. Fenner, one of the new directors to represent active NACE members, is a corrosion specialist for Monsanto Chemical Company, St. Louis, Mo. He has been production supervisor, area supervisor, project engineers, operations research engineer and group leader with the firm since joining it in 1934.

His many NACE activities include participation in organization of the Greater St. Louis Section, serving as the section's chairman, general chairman of the 1957 NACE Annual Conference, chairman of the 1959 Plastics Symposium, chairman of the 1960 Chemical Industries Symposium and also serv-

(Continued on Page 66)



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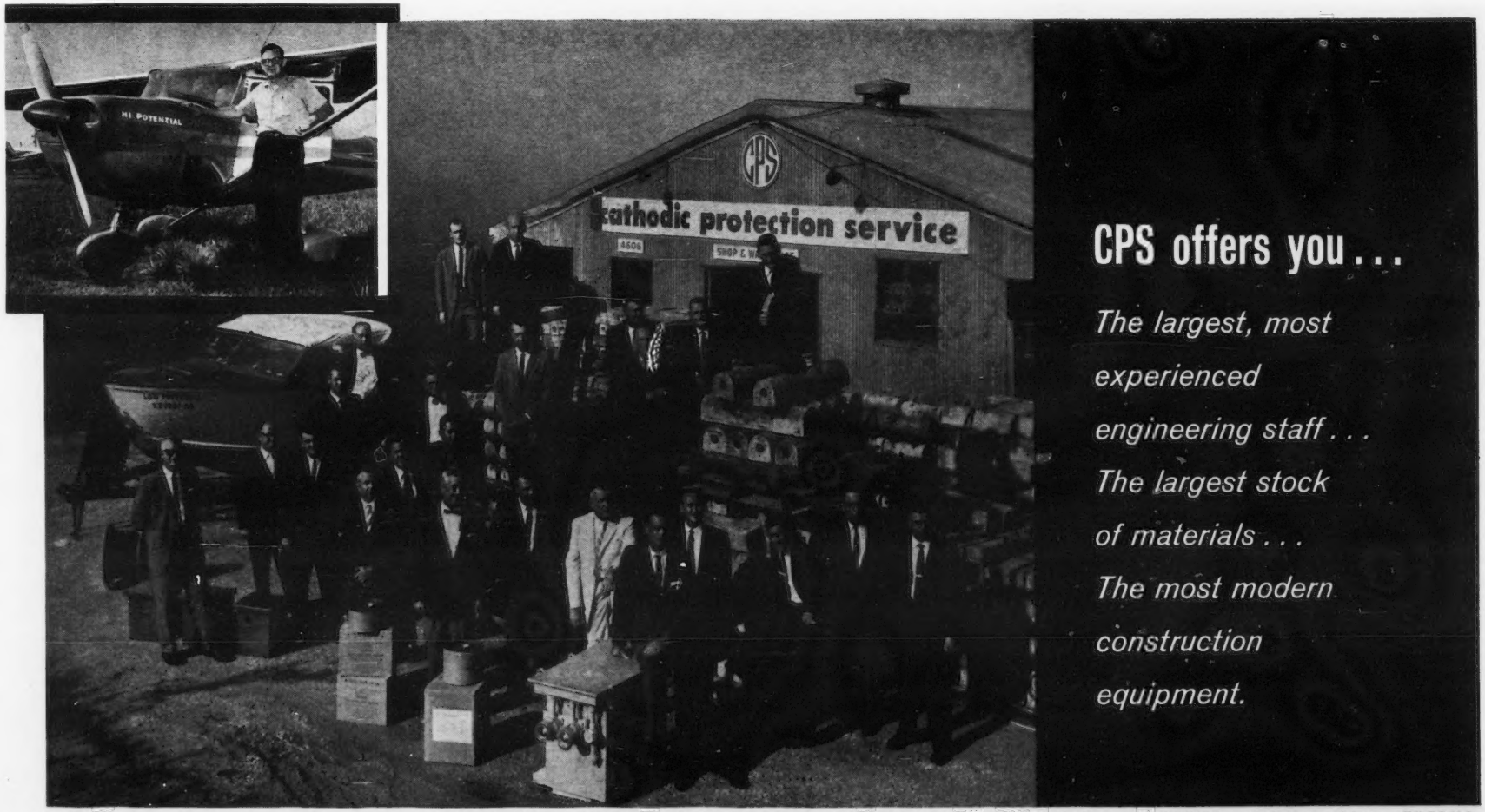

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New Officers and Directors

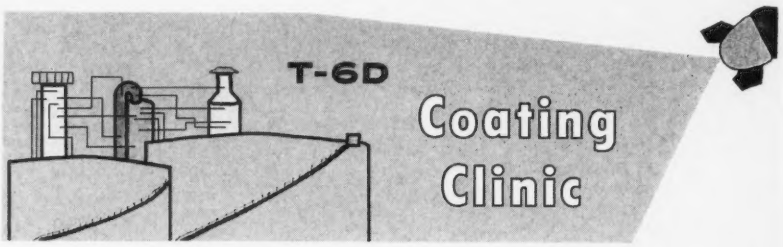
(Continued From Page 64)

ing as a member of the CORROSION Editorial Review Committee. Too, he has served as chairman of Technical Committee T-5D, Plastic Materials of Construction, chairman of Technical Committee T-6, Protective Coatings, and NACE liaison representative to the Society of Plastics Engineers. In addition, he has been a speaker at NACE short courses on corrosion at Ohio State University and Washington University. He holds membership in the American Chemical Society and the Society of Plastics Engineers.

M. E. Holmberg

M. E. Holmberg, also elected to represent active NACE members, joined NACE in 1945. In addition to being the first chairman of Technical Committee T-8, Refining Industry Corrosion, he has actively served on Technical Committees and Task Groups T-1F, T-4F, T-4F-1, T-5B, T-5C-1 and T-5E.

Since 1951 he has operated his own consulting firm in Houston, working on metallurgical and corrosion problems related to the oil, gas and petrochemical industries. Previously he was associated for 15 years with Phillips Petroleum Company in Bartlesville, Okla., during which time he became manager of the company's Test Division. He holds a B.S. in metallurgy from Pennsylvania State College. His memberships in other profession groups include the American Society of Metals, American Welding Society, American Society for Testing and Materials and the American Petroleum Institute.



If you have a question or problem related to industrial paint applications, NACE Technical Unit Committee T-6D will forward it to qualified men in the industrial coating field for answers and suggested solutions to problems. State your problem fully. Questions should be addressed to T-6D Coating Clinic, Corrosion Magazine, 1061 M & M Bldg., Houston 2, Texas.

Sandblasting

Maintaining Nozzle Pressure

What are recommended maximum distances from air supply to sandblast machine and from sandblast machine to nozzle to maintain adequate nozzle pressures for efficient and economic sandblasting?

The problem in most plants and factories that prevents the air compressor of air supply from being close enough to nozzle or point of effective use is a costly and serious one and should be solved as follows:

1. Nozzle diameter should be given first consideration for amount of volume and pressure the nozzle will use when new and when it is worn to the point of capacity of the equipment system being used.

2. The air hose and sandblast hose should be at least three times the nozzle size, that is, ¼-inch nozzle on a minimum ¾-inch ID hose.

3. Air supply valves on air receiver and sandblast machines should be same as full ID hose sizes.

4. Air and sandblast hose coupling ID should be same size as minimum ID hose size.

The above basic considerations hold true when distance from air supply to nozzle is 200 feet—100 feet of air hose and 100 feet of sandblast hose—plus a small 10 to 15-foot tail line or whip hose for operator convenience and efficiency.

Beyond this distance and up to 300 feet, one and a half the recommended internal diameter hose size should be used; gradual reduction of sandblast hose can be made near the nozzle end.

Distances in excess of 300 feet should have portable or temporary air line, such as lightweight industrial aluminum piping (4-inch ID) to furnish air to sandblast machine through 1¼-inch ID valves and air hose or a temporary air line of sufficient size to provide constant flow and pressure acting as an extended air receiver.

When encountering conditions that require end use of nozzle at extreme distances from air supply, extra consideration and planning should be exercised to attain maximum efficiency for a profitable and economical operation.

Sandblasting Steel

What are average consumption figures for specification blasting sand to attain a white metal blast (No. 1 blast) on steel with the following conditions: (1) Heavy rust with deep pitted steel, (2) Light rust with some pitting and (3) New steel, mill scale intact?

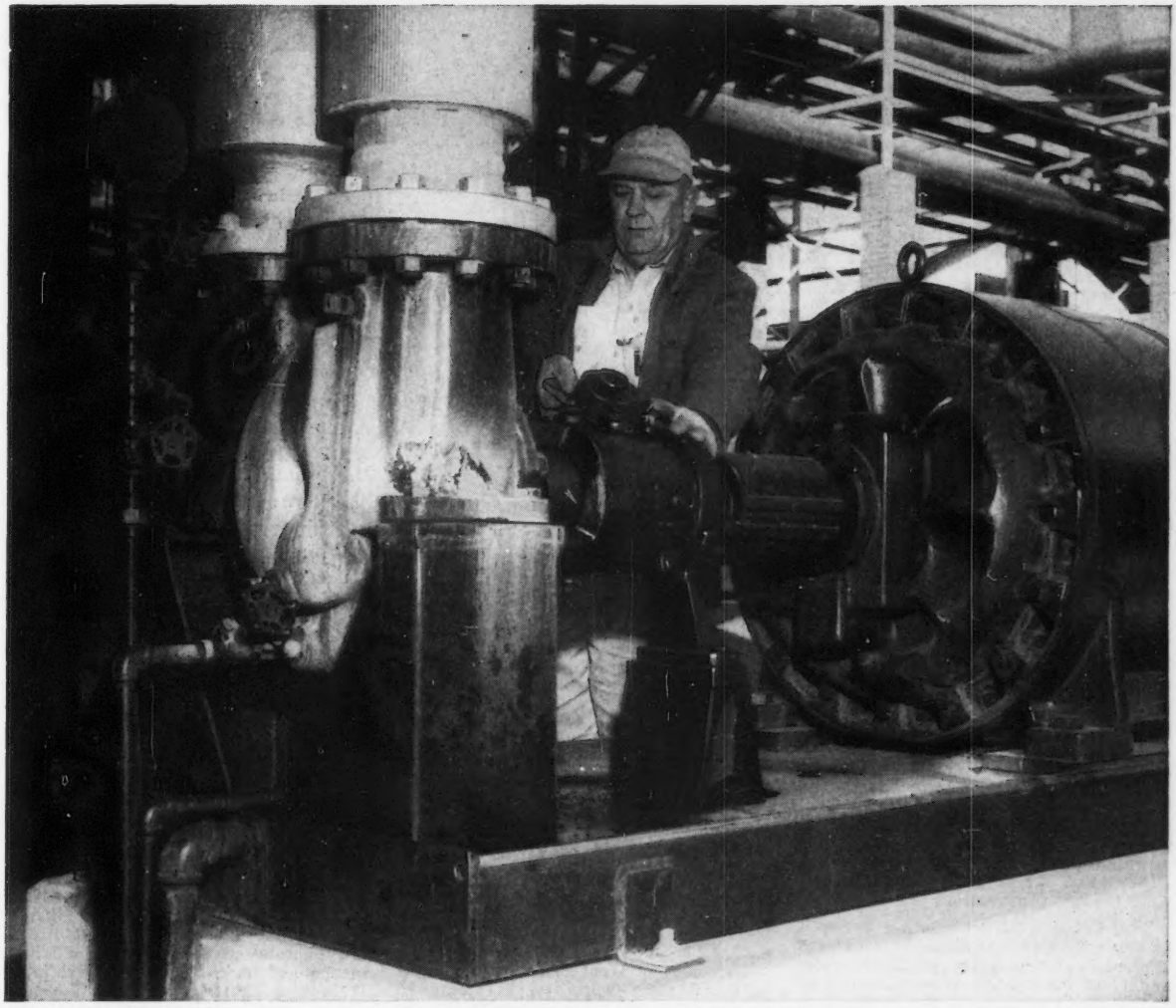
Assuming that a properly balanced sandblasting equipment setup is being used (one consisting of air supply of adequate pressure and volume for nozzle orifice being used, air supply hose and valves of sufficient internal diameter size, modern production type sandblast pressure tank, sandblast hose and couplings of sufficient internal diameter size and tungsten lined nozzle that will maintain constant size), the following amounts of specification silica blasting sand will be used:

Condition 1—Eight to ten pounds of sand per square foot of surface.

Condition 2—Six to eight pounds of sand per square foot of surface.

Condition 3—Four to six pounds of sand per square foot of surface.

An additional comment would be that regardless of size of nozzle being used, the sand consumption per square foot of surface would be the same as long as the blast pattern is directly on the surface being cleaned and is not being lost to overspray.



2½% nickel cast iron impeller in this pump has handled salty crude oil twenty years at Continental Oil's Ponca City, (Oklahoma), installation. Salt concentration varies from 1-10 lbs. per 1000 bbls. handled, at temperatures from 200°-240°F.

2½% Nickel Cast Iron impeller handles briny oil 20 years—without downtime for repairs

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CORROSION ABSTRACTS

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CORROSION ABSTRACTS

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2. TESTING

2.3 Laboratory Methods and Tests

2.3.9, 6.3.2, 3.4.9

Study of Metal Corrosion Using the Heavy Isotope of Oxygen. Pt. II. Corrosion of Cadmium in Humid Atmospheres. A. S. Fomenko, T. M. Abramova and I. L. Gankina. J. Phys. Chem. (Zhur. Fiz. Khim.), **33,** No. 10, 2249-2252 (1959). (In Russian.)

The O^{18} isotope distribution between water, gaseous O, and Cd oxides was studied in the corrosion of Cd in water and O of different isotope composition. In 3% NaCl solution in the presence of gaseous O, corrosion proceeds according to electrochemical and chemical mechanisms. After 30 hours there was no O^{18} isotope exchange between $Cd(OH)_2$ and water, whereas under the same conditions equilibrium was established for O exchange between $Cu(OH)_2$ and water.—MA. 20100

2.3.7, 5.3.4

Possibilities for the Testing of Sprayed Metal Coatings: Proposals for the Unification of Testing Methods. E. Kretzschmar and T. Drazkiewicz. Schweisstechn., **9,** Nos. 1, 2, 11-13, 53-58 (1959).

Available testing methods, originally intended for compact materials, should use for sprayed coatings specimens whose form is adapted to the peculiarities of the material, viz. lack of mechanical bond between coating and base unless heat-treated, and heterogeneity, porosity, and high local hardness of the coating. Shop tests for adhesion, uniformity of thickness, and hardness ("Duroscope" readings correlated with Brinell or Rockwell values) are described, with notes on the spark test, visual control, and penetration tests for d. Recommendations on laboratory tests include the data to be reported, and full details are given of metallurgical examination, hardness specimens and tests, the shear test for adhesion, tensile testing of coating, penetration/time test for d, X-ray examination and assessment of films, wear, corrosion, and fatigue tests. —MA. 19939

2.3.9, 3.7.4, 3.7.3, 3.5.8, 3.5.4

Field Ionization and Field Ion Microscopy. Erwin W. Muller. pp. 154-177 from "Advances in Electronics and Electron Physics," Vol. XIII, 1960, Academic Press, New York, New York.

The uses of the techniques of field ion microscopy in fundamental studies of adsorption and corrosion phenomena as well as in metallurgical research basic to corrosion such as studies of vacancies, interstitials, dislocations, impurities, slip, cold working, strain release, fatigue radiation damage and field-induced chemical reactions are described. —BTR. 21481

4. CORROSIVE ENVIRONMENTS

4.2 Atmospheric

4.2.3, 6.2.5, 7.1

Corrosion of Heat Resistant Materials in Combustion Turbines. V. Cihal, P. Grobner and P. Hoch. Strojirenstvi, **10,** 51-54 (1960) Jan. (In Czech.)

Various heat resistant materials containing 3.45-76.7% nickel, 13.22-26.30% chromium and up to 65% iron are corrosion tested in an air-steam-CO_2-SO_2 atmosphere and at 600-850 C. Corrosion as a function of composition. 13 references.—RML. 20278

4.2.7, 6.2.5, 2.2.7, 3.2.2

Corrosion of Metals in Tropical Environments. Part 5. Stainless Steels. B. W. Forgeson, C. R. Southwell and A. L. Alexander. Naval Research Lab. Rept. 5517, Sept. 19, 1960, 24 pp. Order PB 161749 from Office of Technical Services, Washington, D. C.

Over a period of 8 years, six stainless steels have been exposed to underwater and atmospheric environments in the Panama Canal Zone. Stainless steels are severely pitted during sea water immersion; the mean tide immersion produced one-fourth to one-tenth the pitting of continuous immersion, and the metals were practically unattacked when submerged in fresh water. Greater pitting attack in tropical than temperate seas may be the result of heavy marine fouling. The type and extent of pitting attack of stainless steels is compared with phosphor bronze and several structural steels. Stainless steel is not recommended for sea water service where perforation of structure is a consideration; it was inferior to phosphor bronze and to the structural steels. In fresh water, stainless steels are superior to ordinary steels and equal to phosphor bronze which is noncorrosive in this environment. Tropical atmospheres appear to be more corrosive to stainless steel than temperate climates; the marine effects are slightly greater than the inland. However, precautions should be taken to eliminate cracks where passivity-destroying agents could accelerate pitting attack. (auth)—GRR. 21329

8. INDUSTRIES

8.1 Group 1

8.1.4, 5.4.8, 6.6.5, 3.3.4

Sewer Corrosion and Protective Coatings. C. G. Munger. Amercoat Corp. Civil Eng., **30,** No. 5, 57-59 (1960) May.

Reviews mechanism of corrosion of concrete water and sewer pipe and structures, conditions causing this deterioration, and role of anaerobic and aerobic bacteria. To be effective on concrete, coating must not only have outstanding adhesive qualities and resistance to sewage conditions, but also sufficient thickness to cover imperfections in concrete without pinholing. High polymer vinyl chloride resins are best suited. They are unaffected by oxidation, water, oils, greases, acids, alkalies, salts and bacteria, and can be used as solvent-applied coatings or molded, formed and extruded into flexible or rigid sheets. Sheets can be cast directly in concrete structures and held with mechanical lock. Varied uses are listed. It is now protecting over 7 million square feet of pipe and structures.—INCO. 20352

8.1.1, 5.7.1

Air Treatment. Prevention of Corrosion in Storage. Pts. I, II. L. Walter, Corrosion Technology, **6,** Nos. 11, 12; 327-329; 372-373, 382 (1959) Nov., Dec.

First part discusses properties of air, and reviews various air treatments necessary for preventing corrosion of metal in storage (space ventilation, heating or cooling, dehumidifying, exhaustion of fumes, air filtration, and air conditioning). Second part discusses operation of the different systems in greater detail, and considers approach made by designer of air-conditioning system after basic requirements have been specified. Diagrams.—INCO. 20292

8.1.4, 5.4.5, 5.2.1, 6.6.5

Corrosion in the Water Supply Industry. M. A. Simmonds. J. Inst. Engrs., Australia, **32,** 57-64 (1960) March.

No single item or section of the water supply industry is free from corrosive attack, either from the inside or out. Prevention can best be achieved by a combination of structural design (metal combinations), protective films (paint coatings), alteration of environment (chemical treatment) and cathodic protection. The corrosion problems of concrete are mentioned. 16 references.—RML. 21015

Abstracts in This Section are selected from among those supplied to subscribers to the NACE Abstract Punch Card Service. Persons who are interested in reviewing all available abstracts should write to NACE for information on this service.

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3 Light weight Equipment of ATLAC 382 is generally lighter than wood, Transite, or metal equipment. In one case, a 1,000-gallon tank of ¼" ATLAC 382 weighed 300 pounds, compared with 4.3 times that weight for alloy steel.

4 Economy ATLAC 382 equipment generally costs one-half to one-third less than rubber-lined or tiled equipment, and is normally equal to 304 and lower in price than 316 stainless steel. It is almost always a better buy than the more exotic metals if temperatures and pressures are not too high.

5 Design flexibility There are very few practical limits in size and shape for equipment of ATLAC 382. What's more, equipment can sometimes be redesigned to take advantage of ATLAC 382's unique structural characteristics—resulting in extra simplification, practicality and cost reduction.

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Topic of the Month

Passivating Effect of Elemental Sulfur On Steel in Alkaline Pulping Liquors*

By WALTER A. MUELLER*

Abstract

Sulfur was added to alkaline pulping liquor and the time to passivation measured in two 3260 cu ft kraft digesters. The first digester was made of carbon steel partially coated with a stainless steel overlay while the second was of carbon steel only. With a sulfur addition of 0.32 lb per cu ft of liquor, complete passivation was obtained in both digesters. Without sulfur, passivation was delayed for 72 minutes in the carbon steel digester. The overall results showed that sulfur addition, either as such or combined with anodic protection, is another method of preventing corrosion in alkaline pulping digesters.

5.8.2, 6.2.3, 4.3.3

Introduction

THE RECOVERY OF NaOH from kraft waste liquors has been found to be more efficient than that of sulfur. Thus the sulfur content of the liquor reconstituted from recovered chemicals decreases with increasing efficiency of the recovery. Elemental sulfur can be added to white liquor in order to maintain the required concentration. Dissolution of sulfur in white liquor produces polysulfides and thiosulfate. In an alternative procedure sulfur is dissolved in black liquor, burned in the recovery furnace, and recovered as Na_2S in white liquor. Thus the choice of the method of replenishing the sulfur lost in the recovery varies the polysulfide content of the white liquor.

It has been found from field and laboratory studies that the corrosiveness of white liquor to steel increases with the content of $Na_2S_2O_3$ and of Na_2S_n. However, there is some evidence that the liquor passivates steel when its content of Na_2S_n and $Na_2S_2O_3$ is increased beyond a certain value. Low corrosion rates in digesters can be reached either by maintaining Na_2S_n and $Na_2S_2O_3$ at a very low level in the active state or at a sufficiently high level in the passive state. The safe use of sulfur addition for passivation calls for the measurement of potential-time curves in the whole liquor cycle, e.g. by means of calomel cells.

In a recent field study of this effect, potential-time curves have been recorded in two kraft digesters of 3,260 cu ft volume. The first digester was coated with 310 stainless steel welding overlay, except for the cone, which consisted of the original carbon steel. Electrode No. 1 was introduced in a carbon steel area of the cone and electrode No. 2 above the strainer in an area completely coated with 310 stainless steel. The second digester was entirely of carbon steel and electrode No. 3 was placed in the cone. For the first experiment (Figure 1) 0.32 lb sulfur per cu ft liquor was added to the white liquor (Table 1). No sulfur was used in the second experiment.

A few typical potential-time curves are given in Figures 1 and 2. With sulfur ad-

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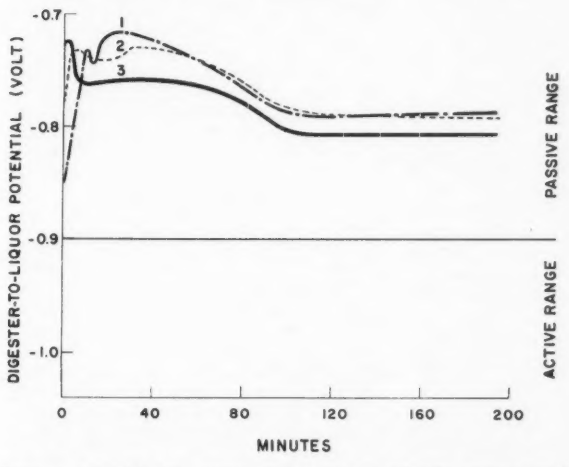

Figure 1—Potential time curves recorded during indirectly steamed cooks (after 0.32 lb sulfur per cu ft was added to the white liquor). Curve 1 was recorded in the cone (carbon steel) of digester No. 1, curve 2 above the strainer (310 stainless steel) of digester No. 1 and curve 3 in the cone of the carbon steel digester No. 2.

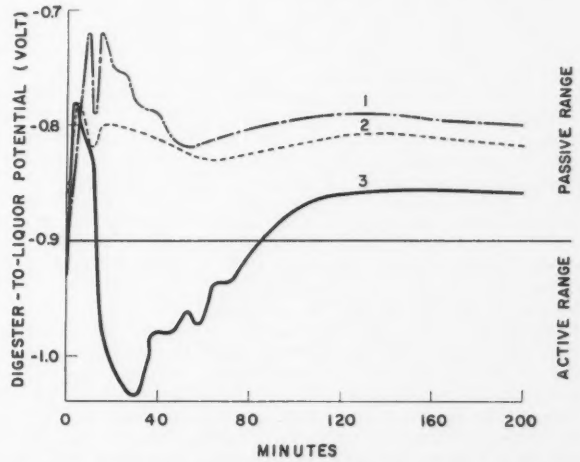

Figure 2—Potential time curves recorded during indirectly steamed cooks without sulfur addition. Curve 1 was recorded in the cone (carbon steel) of digester No. 1, curve 2 above the strainer (310 stainless steel) of digester No. 1 and curve 3 in the cone of the carbon steel digester No. 2.

TABLE 1—Liquor Data

Digester No.	White Liquor Composition			Liquor Volume Cu. Ft.			Percent Black
	Na_2O Eff. Lb./Cu. Ft.	Sulfidity, Percent	Sulfur Addition Lb./Cu. Ft.	White	Black	Total	
2	5.26	28.2	0.32	860	415	1275	32.6
1	5.26	28.2	0.32	835	740	1575	47.0
2	5.03	25.6	0	925	300	1225	24.5
1	5.03	25.6	0	905	620	1525	40.6

dition complete passivation was reached in both digesters. Without sulfur addition, the carbon steel digester was in the active state for 72 minutes; in the first digester coated partially with stainless steel the areas around the electrodes were still passive, but the shift of the potentials closer to the active state suggests that some other area might have been active and corroding rapidly. The similarity between the curves of Figure 1 and those recorded during anodic protection is evident and was expected. Sulfur addition, either as such or combined with anodic protection, is another method of preventing corrosion in alkaline pulping digesters.

Acknowledgment

The author is indebted to the Marathon Corporation of Canada Limited for its generous cooperation in making the mill experiments possible.

Any discussion of this article not published above will appear in June, 1962 issue

Cathodic Protection in Desert Soils*

By W. C. R. WHALLEY

Abstract

An appraisal is made of special conditions contributing to the severe corrosion experienced on buried pipelines in the arid desert soils of Syria and Iraq. The effectiveness of cathodic protection in reducing corrosion in such areas is discussed in detail.

Generally cathodic protection is difficult in desert soils and results are less satisfactory than in moist low resistivity soils. Potentials are notably erratic and are subject to variations of 0.5v within a distance of a few feet. General potential checks at intervals of 200 ft show many points with low potential on pipelines which are polarized (—0.85v or more). In practice the amount of current which can be drained from an element of bare surface is controlled in desert soils by the resistivity of the matrix. Where the bare element is in contact with the high resistivity matrix only, it is possible to drain sufficient current to polarize the pipe surface, provided that the bare element is not too large. Where the bare element is in contact with a lower resistivity salty patch, which is itself enclosed in an envelope of the high resistivity matrix, then it may be a practical impossibility to drain sufficient current through the envelope to polarize the pipe surface. This results in low potential locally.

In unfavorable conditions the general polarization of pipe surfaces in desert soils may be very difficult unless the pipe has first class coating surrounded by substantial thickness of soil or selected ditch padding. Even so, areas of locally low potential may exist. 5.2.2, 4.5.2

About the Author

W. C. R. WHALLEY received his MSc degree from the Chemical Engineering Imperial College, London, England, in 1937. The same year he joined the Iraq Petroleum Company where he worked pipe line operations throughout the Middle East. In 1945 he was appointed Senior Corrosion Engineer for the IPC.

Introduction

THE PRINCIPAL oilfield in Northern Iraq extends from the town of Kirkuk some 70 miles northwards toward Mosul. The present production is 600,000 bbls per day. Pipelines connect the field with the oil ports of Banias and Tripoli on the Mediterranean Coasts of Syria and Lebanon respectively. The first lines (12-inch) were completed in 1933. During the past 12 years, 16-inch and 30-inch lines may have been added in the same right-of-way. Extensive looping of the 30-inch is at present in hand and capacity is being steadily increased.

Figure 1 shows a sketch map of the area. For a large part of the right-of-way the pipelines traverse the deserts of Northern Iraq and Syria. Kirkuk field is near the western edge of the Kurdish mountains, which lie along the border between Iraq and Persia. The field itself is in a fertile zone of 15-inch rainfall. Proceeding westward, however, rainfall diminishes sharply and desert conditions prevail over the greater part of the alignment, until reaching a region of Syria, approximately 70 miles inland from the Mediterranean coast line.

The coastal ranges of Syria and Lebanon cast a rain shadow over the Syrian desert. Generally speaking the rainfall in the area is around 5 inches annually. The profile of the line is closely related to the rainfall, Figure 2. The coastal regions bordering the Mediterranean enjoy a high rainfall on the seaward or western side of the mountains. The land is fertile and villages numerous. Passing inland, however, the peaks of the ranges mark the limits of high rainfall and fertile ground. Eastward, on the inland side of the coastal ranges, desert conditions are quickly reached once the watershed is crossed, and agriculture is carried on only where irrigation is possible.

Soil Corrosivity

In the Middle East the quantity of rainfall marks a critical distinction between corrosive and more inert soils. It is the truly arid desert soils that receive a rainfall of less than six inches, which are generally corrosive. In the coastal areas of high rainfall, corrosion is strictly local in character and usually associated with marshes or damp clay. These often contain sulfate-reducing bacteria. Considered as a whole, however, the soils of the well-watered coastal plain are not seriously corrosive. Along the edge of the desert is a belt of dry but cultivated ground with rainfall 8-12 inches. This too is substantially inert. Generalized corrosion begins quite sharply at the edge of the desert and coincides with the limit of dry farming. In Middle East conditions this latter limit occurs at about eight inches rainfall.

A characteristic feature of these desert soils is their lack of homogeneity. A multiplicity of low resistivity salty patches lie scattered throughout a matrix of high resistivity ground. The typical desert soil receives insufficient annual rainfall to carry soluble salts deep into the earth. Where the rainfall exceeds 10 inches, these is a penetration into the sub-soil and the rain flows down deep into the earth, taking with it any soluble salts which it may have leached out of the surface layers. Solubles that remain near the surface are dispersed throughout the soil.

In these desert areas east of the coastal hills, however, where the usual annual rainfall is less than 6 inches, the soil is not leached in this way by the brief winter storms. The short but possibly violent rainstorm is followed by sunshine and drying winds. The surface is quickly dried and reverse capillarity brings the moisture again to the surface, before it has had time to penetrate deep into the ground. This alternation of short downpours with sunshine and drying winds has the effect of concentrating the soluble salts already present in the soil into restricted areas. These local concentrations form saline patches when the ground dries out.

A great number of salty patches is a typical feature of desert country, wherever the soil is of a clayey nature with some powers of water retention. Where the surface consists, however, of loose sandy particles with small water-holding power, reverse capillarity is not effective and a small annual rainfall suffices to leach out the surface layers. Further evidence that the winter rains do not penetrate deeply into the ground in these desert soils is afforded by the search for water-bearing formations. The neighborhood of great mud flats which become lakes after heavy rain is not a favorable site for drilling water wells. An area of barren rocky hillsides where the soil cover is insufficient to obstruct the seepage of rain into the ground is a more likely prospect for a water well.

Throughout the alignment of the pipelines the surface is usually composed of a light clay or marl. Sand dunes are exceptional. The soil generally is plentifully dotted with salty patches. Each patch may extend from a few inches to a few yards. In extreme cases where a closed basin exists, salt marshes or lakes may be formed. In the neighborhood of the Wadi Tharthar in Northern Iraq and of Palmyra in Syria are typical closed basins where an extensive area of desert drains into a salt marsh with no outlet to the sea.

The movement of the soluble salts in the soil may be quite rapid. The author is acquainted with an area of semi-desert which was brought under irrigation in 1939. At that time the ground appeared quite salt free, was fairly high in resistivity and well-vegetated in the rainy season. During the course of the following few years the various hollows and basins in this area were transformed into encrusted salt pans resulting from drainage from the nearby irrigated land.

The presence of small saline patches

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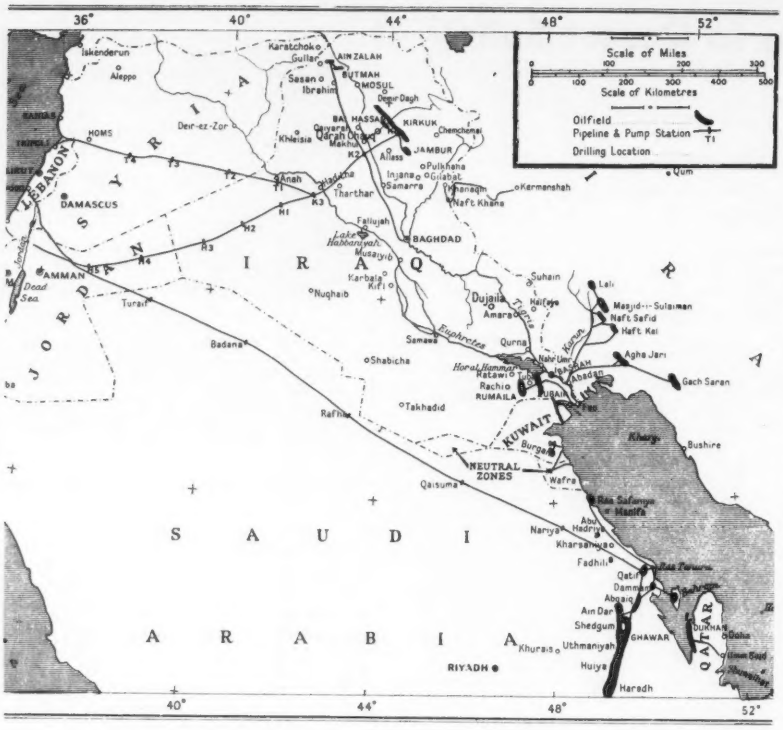
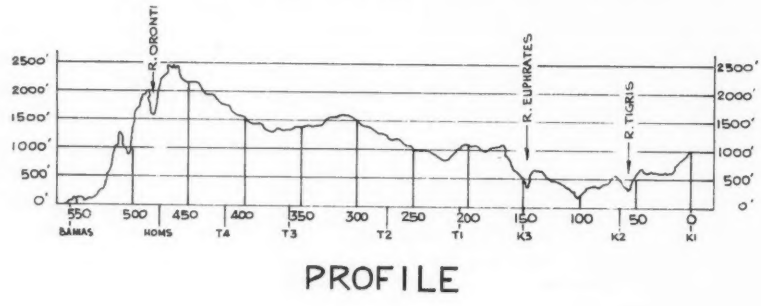


Figure 1—Sketch map of Middle East oilfields, pipe lines and drilling locations.



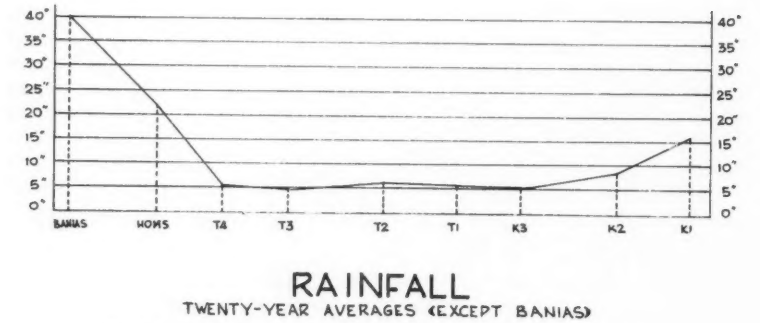


Figure 2—Cross section of altitude and proportionate rainfall at key points.

can often be detected by eye and confirmed by soil resistivity tests or analyses. Chlorides, sulfates, and occasionally nitrates, of sodium, magnesium and calcium are the usual constituents. These patches tend to be deliquescent and retain moisture long after the surrounding soil has dried out. Thus resistivity, moisutre content and oxygen content vary sharply in soils where salty patches are numerous. It must be emphasized that these saline patches exist in vast numbers, scattered throughout the desert area. To their presence is undoubtedly due the paradox that a relatively rainless area should be highly corrosive. The extent of individual patches is measured by a few cubic inches or a few cubic feet in the majority of cases.

The correlation between rainfall, fertility of the ground, presence of saline patchs, and corrosion of buried pipelines, is clear and sharply defined in Middle East conditions. It is a common observation that if the local winter rainfall is sufficient to support a crop of barley without irrigation, then the soluble salts in the soil are dispersed, not concentrated into patches, and the soil is not corrosive to a serious extent. This observation has been repeatedly verified along the fertile fringe of the desert. Here the presence of a range of hills may lead to sharp rain shadows. Within a distance of a few miles the local rainfall may fade from 12 to 14 to 6 to 8 inches without any obvious change in the nature of the soil. Experience shows that cultivation is a good guide to the incidence of future corrosion. Attention should be concentrated on the infertile soils beyond the limits of cultivation by dry farming. Such considerations do not, of course, apply to irrigated regions or the neighborhood of brackish springs which occur in places along the fringe of the desert.

Regarding the resistivity of desert soils, the matrix generally has a high (10,000 to 50,000 ohm cms) resistivity in summer conditions. The innumerable salty patches show decided lower resistivities of 500 to 3,000 ohm cms. In general the surface layer of fine soil is thin with more or less consolidated rock at depths of one to three feet. All pipelines are buried and in the case of the 30-inch line the depth of the ditch is 5 ft. The disturbed ground of the backfill does not consolidate to the consistency of the surroundings, but remains loose even after 20 years. This results in the pipe ditch acting as a drain for the surrounding ground and soluble salts tend to accumulate in the ditch. Soil temperatures at pipe depth range over the year from 55 F to 80 F. Soil moisture is difficult to measure but is believed to be low in the desert areas for the greater part of the year.

Cathodic Protection

Cathodic Protection began in 1946 and was extended to include the pipeline system generally by 1953. The power supply is by means of an 11 kV single phase aluminum sheathed cable suspended on the poles of the telephone system. The cable is energized from the pump stations. Rectifier sub-stations are spaced at intervals usually of five to seven miles, but less in some places. A stepdown transformer supplies a rectifier. The standard rectifier is an oil-cooled 50 v 40 amp type. Where additional current is required two or more are used.

The heaviest current consumers are the earlier 12-inch and 16-inch lines, taking 3 to 5 amps per mile on the average. The more recent 30-inch lines were well-coated, using a double coat of C.T. enamel with fiberglass and asbestos or kraft outer paper. The current require-

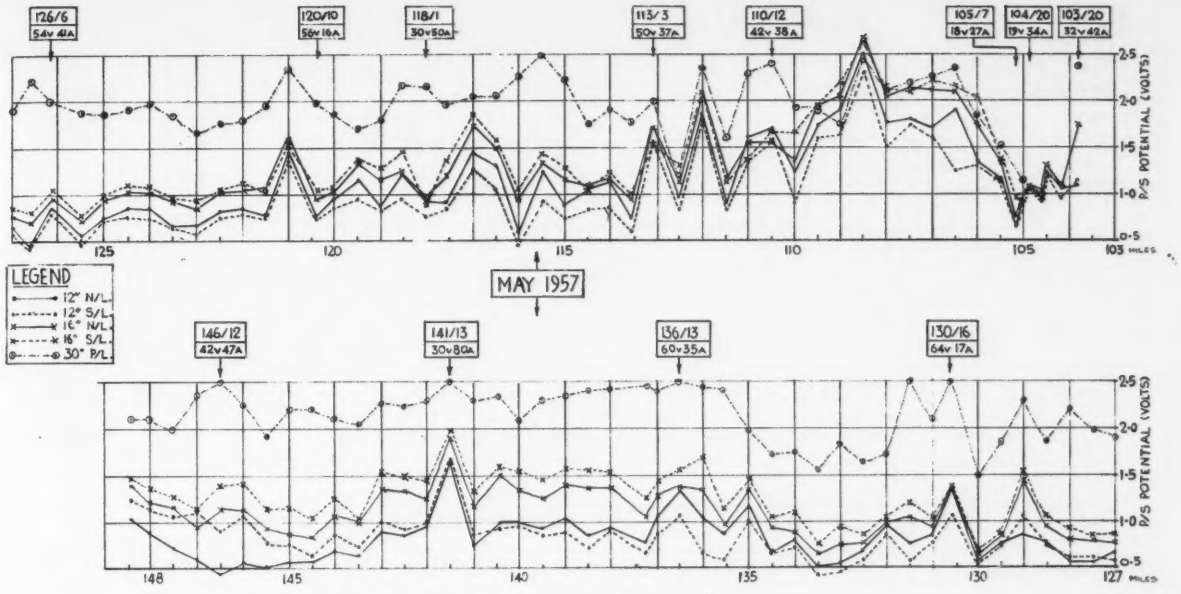


Figure 3—Erratic potentials at ½-mile intervals.

ments of the 30-inch lines are small, usually from 0.2 to 0.5 amps per mile.

Soon after the first application of cathodic protection, it was noticed that pipe potentials on the poorly coated 12- and 16-inch lines were not satisfactory, and these have continued to deteriorate. On the well coated 30-inch lines, potentials generally are better. The unsatisfactory character of pipe potentials takes several forms. The spread or cover obtained from each installation has contracted in many cases so that stretches remote from the nearest rectifier are below —0.85 v in spite of increased current. This is, of course, a common feature of cathodic protection systems and attributable to coating deterioration.

What has caused more concern is the peaking and erratic changes of potentials as between adjacent observation points, Figure 3. Such erratic and abrupt changes lead to distrust of potentials at points intervening between successive observations. Originally the system of parallel pipelines was intended to be treated as a single structure, with the various parallel lines bonded together at intervals of one or two miles. Later experience called for the increasing of the number of bonds, particularly in the vicinity of the rectifiers. Later again, however, it was concluded that it would be better to treat the lines as isolated structures. Bonds have been disconnected except at the rectifier sub-stations.

Observation points were provided for potential measurements at one mile intervals in the first instance. Experience of erratic potentials between adjacent observations promoted the reduction of the spacing to half-mile intervals, with additional observation points approximately 200 ft on either side of each rectifier. The half-mile observations revealed many points of low potential not previously known. Subsequently potentials were taken at 200-ft intervals or less over the entire system. This was done without damage to the pipe coating by attaching a long wire to the nearest permanent observation point and moving the electrode along over the top of the pipe. In some sections this examination showed highly erratic potentials on the poorly coated 12- and 16-inch lines, with variations of ½ v or more within a distance of 200 ft. (Figure 4.)

Still closer examination at 15-ft intervals revealed that in this variable desert environment soil potentials may change by ½ v or more within the space of a few feet. Of course, it is the ground potentials which vary. The pipe, being a continuous metallic structure, is substantially at uniform potential over short distances. These erratic differences represent the changes in boundary potential between the pipe and the contacting soil. The author has always regarded the boundary potential as being the critical consideration in assessing the effectiveness of cathodic protection because it is at the boundary where corrosion is liable to occur. Obviously the checking of potentials at 200-ft intervals is a laborous process which could not be repeated frequently.

The usual practice is to place the electrode on the surface of the ground over the pipe. Numerous checks have usually shown only small differences rarely exceeding 0.1 v as between positioning the electrode at ground level over the pipe and in the soil immediately adjacent to it. It has been suggested that in unfavorable soil conditions large potential differences may exist as between the soil at the top and the bottom of the pipe. This might be expected perhaps where a poorly coated line is bottomed in rock. The author has done little in the way of checking potentials at the underside of lines owing to the formidable excavations involved and the danger of damaging the coating. It seems that the presence of ditch padding below and around the pipe should minimize such differences even in rocky ground.

Experience shows that in unfavorable conditions increasing the current drained by the nearest rectifier has little effect in raising low potentials at points intermediate between rectifiers. The installation of an additional ground bed adjacent to the low section is often but not invariably a cure. There is a tendency for additional low potentials to develop where exceptionally heavy currents are employed. It is clear that it is only in cases of reasonably favorable ground conditions that heavy concentrations of current may be usefully employed.

The winter rains have their effect on cathodic protection. Substantial seasonal differences occur both in potentials and spread at constant current output. The author has not been able to trace a general pattern of behavior in such seasonal changes. Ground bed resistances also change but not so drastically as was originally anticipated. Ample evidence of the necessity for frequent potential checks is available. Potential measurement at half-mile intervals are carried out at bimonthly intervals.

In desert conditions the peculiar and special nature of the soil is a dominant factor in cathodic protection. In some locations the soil is deep; in many, however, it is shallow with rock beneath the surface. It is a characteristic feature of these desert soils, as previously stated, that they comprise a high resistivity matrix, speckled with innumerable isolated patches of relatively low resistivity. In some places limited areas of reactive low resistivity clays occur, often associated with leaks.

Effective cathodic protection depends on draining from every contacting element of the pipe surface sufficient current to establish a protective potential at

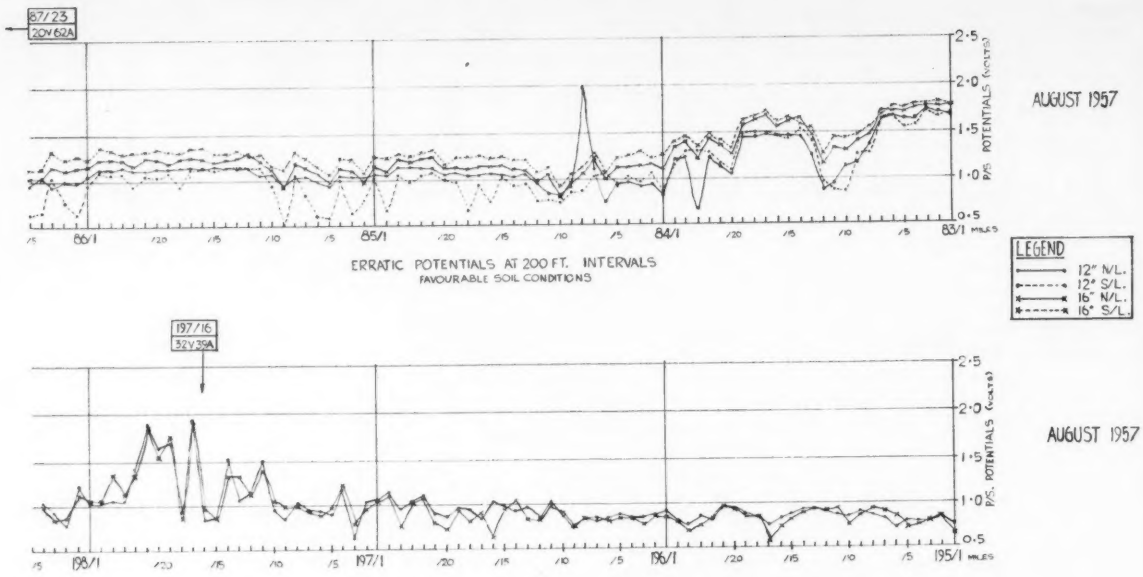


Figure 4—Erractic potentials at 200 feet intervals in somewhat favorable soil conditions.

the boundary between pipe and soil. The current density required is increased in soils of low resistivity and in the presence of a plentiful supply of oxygen. It seems, however, that in practice the amount of current which can be drained from an element of bare surface is controlled in desert soils by the resistivity of the matrix. Where the bare element is in contact with the high resistivity matrix only, it is possible to drain sufficient current to polarize the pipe surface, provided that the bare element is not too large. Where, however, the bare element is in contact with a lower resistivity salty patch, which is itself enclosed in an envelope of the high resistivity matrix, then it may be a practical impossibility to drain sufficient current through the envelope to polarize the pipe surface. It is evident that this difficulty is intensified in the case of shallow soil thinly overlaying rock, especially on the underside of the pipe by restriction of the current path.

Contrariwise, in homogeneous low resistivity soil, or better still in salt water, the difficulty does not arise. The contacting medium is uniform. Such installations have substantially a self-adjusting characteristic, insofar as coating differences are concerned. Current density at the pipe surface distributes itself locally to establish a uniform potential, provided that sufficient current is drained from the structure as a whole.

Regarding the various types of ground conditions, it is possible to distinguish between favorable and unfavorable characteristics as follows:

Ground Conditions Favorable to Cathodic Protection

1. Deep soil (to several feet below pipe depth).

2. Resistivity reasonably low (10,000 ohms or less). Resistivity decreasing with depth is a favorable circumstance. Accordingly the existence of a water table at depths of 20 to 100 ft is favorable, provided it is not insulated from the pipeline by intervening high resistivity strata.

3. Homogeneiety is important. Reasonable uniformity in resistivity and physical character is favorable; for example, a light sandy clay of 5,000 ohms with good depth. Pebbles and scattered rocks are immaterial in such a case. Wind-blown sand is too loose and inert to encourage segregation of salty patches and is quite a favorable medium if it is deep and has a reasonable resistivity (10,000 to 30,000 ohms).

Ground Conditions Unfavorable to Cathodic Protection

1. Shallow soil, thinly overlaying rock, constitutes a difficult case owing to the higher resistivity of the rock. If the rock itself is of exceptionally high resistivity, the difficulty is accentuated. A sub-soil of weathered limestone is less difficult a case than a bed of crystaline gypsum. In rock the pipe ditch acts as a drain and may accumulate soluble salts from the surrounding ground. Solubles and water also move slowly toward the pipe as a result of electrolysis. Where the pipe ditch is bottomed in solid rock, this accumulation is an additional source of difficulty which increases with the passage of time.

2. Where the pipe itself is at shallow depth even in deep soil, this circumstance is unfavorable. The conducting path through the ground is restricted in respect of the top of the pipe.

3. High resistivity soil is unfavorable if it contains patches of low resistivity.

4. Non-homogeneous soil is unfavorable, particularly if the low resistivity portions are local and isolated one from another. The typical desert soil of light sandy clay containing a multitude of unconnected salty patches is unfavorable.

When several unfavorable conditions are combined, effective cathodic protection is hardly possible unless the pipe has a first-class coating. For instance, a shallow salty soil thinly overlaying massive crystaline gypsum in which the pipe lies presents a difficult problem. This is the condition of Figure 5, which may be compared with Figures 6 and 7 in which soil conditions are unfavorable. The state of the pipe coating is the factor which overrides all others in such unfavorable circumstances. In the sections illustrated, a well coated 30-inch pipe lies in the same right-of-way as twin 12-inch pipes and twin 16-inch pipes. Current drainage requirements necessitates that the 30-inch pipe coating contain only infrequent small pinholes by comparison with the older poorly coated 12-inch and 16-inch pipes. Moreover the 30-inch pipe was provided with a 6-inch layer of dirt padding all around it. These advantages tend to outweigh unfavorable soil conditions.

Typical coating characteristics of the lines in question are shown in Table 1.

The considerable difficulty, first in securing a general polarization of the pipe surface, and secondly a checking that every element of the pipe surface is, in fact, polarized, obviously raises questions as to the degree of effectiveness of cathodic protection in desert soils. To this there can be only the one final answer—the incidence or absence of leaks. It was originally hoped that the installation of cathodic protection would result in a cessation of leaks on the old 12-inch lines. This desirable condition would provide a guarantee of their non-appearance on the more recent 16-inch and 30-inch lines. After six years of cathodic protection, leaks on the old lines have not ceased. Nor in view of the many known or suspected low potentials on the older pipes can a cessation be expected. Thus, the anticipated practical check of the effectiveness of our cathodic

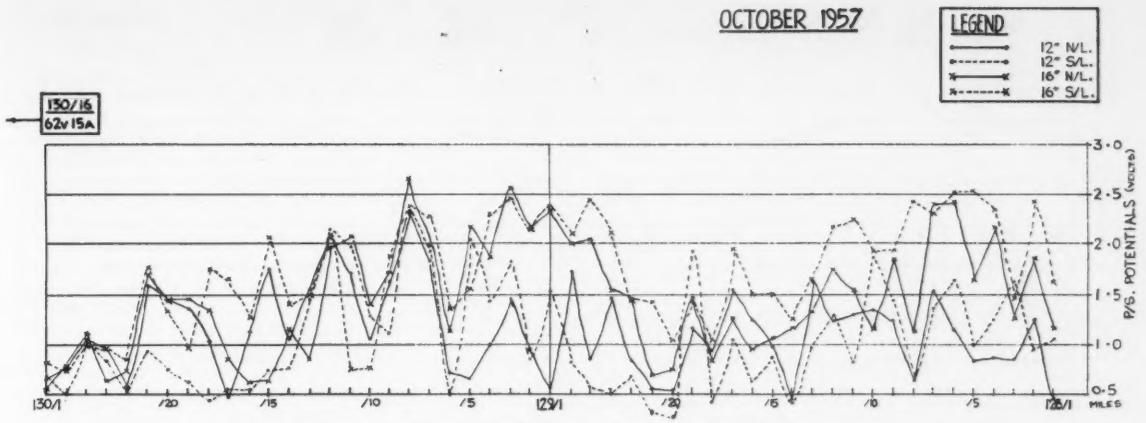


Figure 5—Erratic potentials at 200 feet intervals in unfavorable soil conditions.

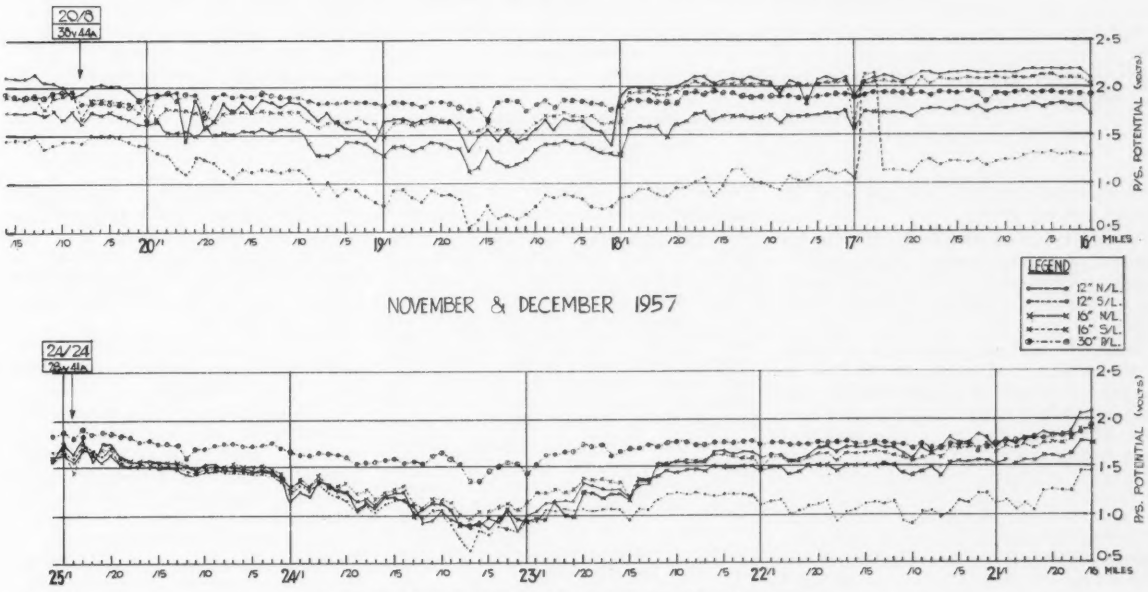


Figure 6—Potentials at 200 feet intervals in favorable soil conditions.

protection has not been obtained. Hypothetical considerations are considered for an appraisal of the effectiveness of cathodic protection on these pipelines. Table 2 shows the total annual leaks for the past 15 years.

Experience shows that large numbers of perforations already exist concealed in the old lines, which may come to light when the line is disturbed. Thus, reconditioning or a change in pressure conditions brings out a plentiful crop of leaks. The record shows a great outbreak in 1957, attributable to emergency pumping conditions. These resulted in high pressures on sections of the line which had not been pressurized for many years.

There is some reason to suppose that the application of cathodic protection to an old corroded line may be expected to result in the loosening of the oxide scale. This action naturally brings to light leaks hitherto dormant. It also results in increased current requirements, since at many points corrosion has spread under the original coating, which thus becomes detached complete with adhering scale. How far the corroding surface beneath such loose scale can be polarized even if sufficient current can be made available is a matter for surmise. In the absence of a conducting medium in contact with the pipe surface, there can be no cathodic protection. It is possible that successive winter rains may convey sufficient soluble salts to the pipe surface to establish a conducting path. Whether such a conducting path will persist throughout the year or be sufficient is open to conjecture. It is clear that in a moist environment such difficulties do not arise, but in desert soils they seem important. A side effect of cathodic protection is the slow concentration of moisture in the immediate vicinity of the pipe. The extent of this movement is not known but its effect must be to increase current requirements in arid ground. The presence of moisture in sufficient quantities should improve contact conditions at the pipe surface. Alkaline liquid is sometimes noticed between the coating and the pipe in cases of poor bond.

From the foregoing it will be seen that the author's original expectations regarding the efficacy of cathodic protection on pipelines in desert soils have been considerably modified as a result of operating experience. The unforeseen difficulties brought to light may be listed briefly as follows:

1. In unfavorable soil conditions general polarization of the pipe surface may be so difficult as to be impracticable, unless it has a first-class coating surrounded by selected dirt as ditch padding. This is an extreme case which applies only to a portion of the right-of-way discussed.

2. In desert soils pipe which is generally polarized may still have areas of low potential due to excessive coating defects or the masking effect of corrosion scale. These low potentials can be detected only by laborious checking. This case is more widespread.

3. Deterioration of the older pipe coatings has proceeded more rapidly than expected, as evidenced by increased cur-

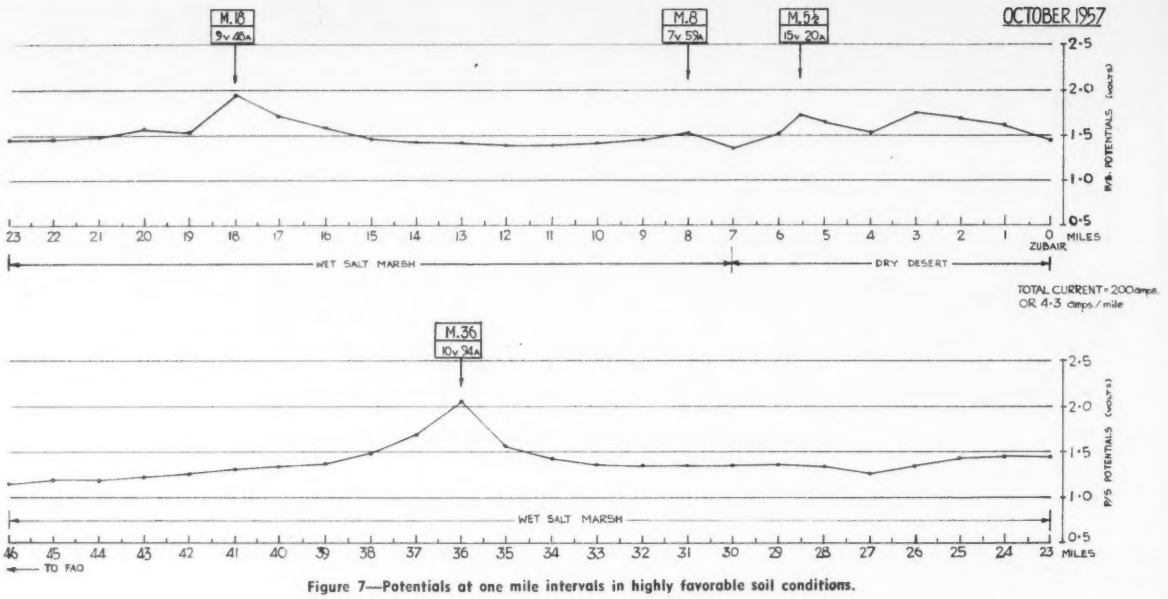


Figure 7—Potentials at one mile intervals in highly favorable soil conditions.

rent requirements and contraction in spread between units.

4. The effect of heavy oxide scale in masking the pipe surface was insufficiently appreciated, as was the tendency for cathodic protection to detach such scale.

From the author's experience in other areas he has good assurance that in a moist environment of reasonably low resistivity, polarization of a pipe surface to —0.85 v will suppress corrosion. Even on a poorly coated old line, with a long history of corrosion, leaks can be halted in favorable soil conditions. In arid desert soils, however, experience to date has been less successful. The question arises how far can one expect cathodic protection to be effective in such special conditions? Recent inquiries in the United States of America indicated that somewhat similar difficulties have been experienced in such extremely arid areas as those of Southern California, where rainfall likewise is around five inches only. This tends to suggest that the author's experience may be typical of desert areas having a rainfall below eight inches.

TABLE 1—Typical Coating Characteristics of Specified Lines

Line Size	Current Drained	Coating Resistance
30-inch line	0.2–0.5 amps/mile	200,000 ohms/sq ft
16-inch line	2–4 amps/mile	4,000 ohms/sq ft
12-inch line	4–10 amps/mile	1,000 ohms/sq ft

Regarding remedies for the difficulties mentioned above, one can conclude that these are essentially a matter of taking action in good time. In all main line construction projects pipe coating should receive great care. First rate materials, careful application, and inspection are essential. In rocky ground the operations of ditch padding and lowering-in must receive continuous attention. The presence of a layer of fine dirt below and around the pipe, not only protects the coating but facilitates cathodic protection in rocky ground. In arid desert soil one cannot expect completely to arrest the process of corrosion once it has begun. Accordingly cathodic protection should be applied without delay following completion of the pipeline.

Conclusions

1. The effective application of cathodic protection is dependent on favorable conditions to a greater degree than was previously realized.

2. The favorable condition of prime importance is a high-class coating. This should be surrounded by a substantial thickness of selected fine soil.

3. The nature of the ground also is a controlling condition and in a favorable environment poorly coated or even bare steel can be prevented from corroding by cathodic protection.

4. Arid desert soils often constitute an unfavorable environment and only in so far as this disadvantage is offset by a high-class coating can cathodic protection be expected to be effective.

5. In desert soils the successful cathodic protection of an already corroded line is a difficult and uncertain undertaking. The screening effect of corrosion scale makes it difficult both to obtain general polarization and to check that it has, in fact, been obtained.

6. The cathodic protection of lines in moist soil of low conductivity is straightforward and effective.

7. In arid desert soils, because it is a difficult and uncertain matter to arrest corrosion once it has started, cathodic protection should be applied immediately following construction of any line.

Acknowledgments

The author wishes to express his thanks to the Management of the Iraq Petroleum Company, Limited, for permission to publish this paper, and to his colleagues on whose collective experience it is based.

DISCUSSION

Comments by L. P. Sudrabin, Consultant to Electro-Rust-Proofing Corporation, Belleville, New Jersey:

The author has made an excellent appraisal of the special conditions contributing to the severe corrosion experienced on buried pipelines in arid desert soils. A good pipeline coating system, able to withstand the temperature, pressure and voltage stresses, is most essential in obtaining the corrosion control required.

In an engineering study of the corrosion control program on large diameter pipelines in arid desert regions, between

TABLE 2—Leaks on 12-Inch Lines, 665 Miles Total Length

Year	1945	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60
Leaks	55	44	24	24	53	73	31	109	80	68	31	85	325	104	60	56
									←CATHODIC PROTECTION→							

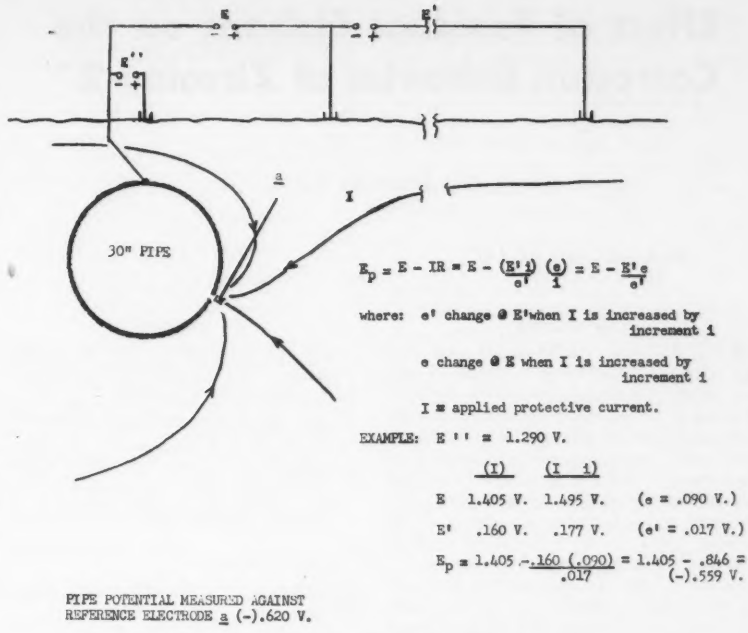
the Persian Gulf and the Mediterranean, the discusser has observed that the pipe potential measured against a reference electrode positioned on the ground surface was not truly indicative of the potential existing at the pipe surface. It was found that a pipe potential of —.85 volt to a $CuSO_4$ half cell positioned on the ground surface, particularly in high "apparent" resistivity soils, does not provide all the corrosion control that can be obtained from cathodic protection.

A representative observation on a poorly coated pipeline in high resistivity desert soil, where severe corrosion has been experienced, is illustrated in Figure 1. An exposed strip, attributable to shoe damage during field coating application, was known to extend longitudinally along a section of the pipeline. The pipe potential measured to a reference electrode positioned over the pipe (E″) and at the flaw (**a**) was —1.290 volts and —.620 volts, respectively. The pipe potential measured to the probe electrode positioned on the well coated surfaces closely approached —1.290 volts.

An approximation of the pipe potentials to a close reference electrode was determined by a modification of a method for IR drop correction proposed by J. M. Pearson.[1] The random disposition of coating flaws and non-uniform soil resistivities will not provide the symmetrical distribution of potential gradients needed by the Pearson Method. Because earth field and stray current gradients were found to be negligible on the pipeline, two matched $CuSO_4$ half cells positioned approximately three feet and 30 feet to one side of the pipeline were used to measure E, E′ and the incremental gradient changes e, e′ attributable to increasing the applied current I by an amount i. In Figure 1, it will be noted that the calculated potential of the pipe (E_p) —.559 volt closely approaches the measured value at (a)—.620 volt.

In Figure 1, most of the IR drop included in the pipe potential measured to a reference electrode positioned on the ground surface cannot be included in the cathodic polarization effect[2] needed to control the local cell short lines of corrosion current flow.

The Trans-Arabian Pipe Line has experienced a marked reduction in the cumulative leak record by reconditioning and recoating the pipeline in aggressive soil areas where poor and nonadherent coatings occur. The recoating work is supplemented by a vigorous cathodic protection program.


E
E'
E''
a
I
30" PIPE
$E_p = E - IR = E - \frac{(E'i)}{e'}\frac{(e)}{i} = E - \frac{E'e}{e'}$
where: e' change @ E' when I is increased by increment i
e change @ E when I is increased by increment i
I = applied protective current.
EXAMPLE: E'' = 1.290 V.
(I) (I i)
E 1.405 V. 1.495 V. (e = .090 V.)
E' .160 V. .177 V. (e' = .017 V.)
$E_p = 1.405 - \frac{.160\ (.090)}{.017} = 1.405 - .846 = (-).559$ V.
PIPE POTENTIAL MEASURED AGAINST REFERENCE ELECTRODE a (-).620 V.

Discussion Figure 1—E_p pipe potential to close electrode.

Question by Tom Watson, Corrosion Service Limited, Toronto, Ontario, Canada:

Are you not sure that the erratic potentials shown were not the effect of varying IR drops between the electrode and the pipe surface in soils and widely varying resistivity?

Reply by W. C. R. Whalley:

Numerous checks show only small differences in such cases between the potential at ground level over the pipe and at pipe depth. Such differences rarely amount to as much as 0.2 v.

Questions by Glenn R. Roberson, Standard Oil Company of Texas, Houston 1, Texas:

1. Is a potential with the electrode on top of the ditch satisfactory or do they have to get closer to the pipe?

2. Was the coating in good condition where they found little difference in the readings?

Replies by W. C. R. Whalley:

1. We found little difference.

2. Yes, in so far as gross defects were not visible.

[1] Pearson, J. M., *Corrosion*, 3, 549, 1947.

[2] Sudrabin, L. P. and Ringer, F. W., *Corrosion*, 13, 351t (1957) May.

Any discussion of this article not published above will appear in June, 1962 issue

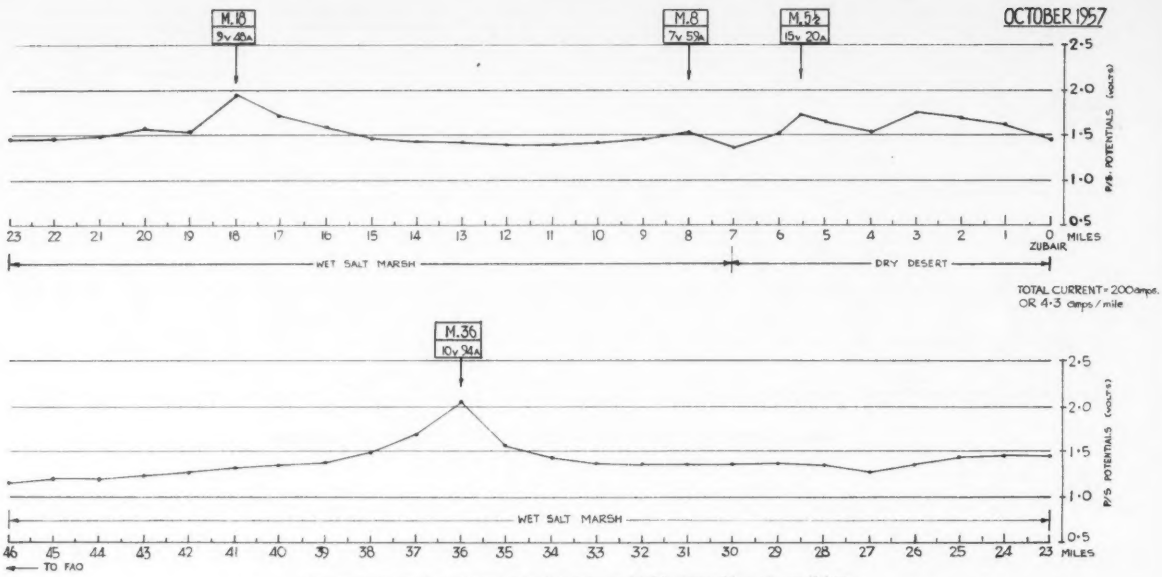


Figure 7—Potentials at one mile intervals in highly favorable soil conditions.

rent requirements and contraction in spread between units.

4. The effect of heavy oxide scale in masking the pipe surface was insufficiently appreciated, as was the tendency for cathodic protection to detach such scale.

From the author's experience in other areas he has good assurance that in a moist environment of reasonably low resistivity, polarization of a pipe surface to —0.85 v will suppress corrosion. Even on a poorly coated old line, with a long history of corrosion, leaks can be halted in favorable soil conditions. In arid desert soils, however, experience to date has been less successful. The question arises how far can one expect cathodic protection to be effective in such special conditions? Recent inquiries in the United States of America indicated that somewhat similar difficulties have been experienced in such extremely arid areas as those of Southern California, where rainfall likewise is around five inches only. This tends to suggest that the author's experience may be typical of desert areas having a rainfall below eight inches.

TABLE 1—Typical Coating Characteristics of Specified Lines

Line Size	Current Drained	Coating Resistance
30-inch line	0.2–0.5 amps/mile	200,000 ohms/sq ft
16-inch line	2–4 amps/mile	4,000 ohms/sq ft
12-inch line	4–10 amps/mile	1,000 ohms/sq ft

Regarding remedies for the difficulties mentioned above, one can conclude that these are essentially a matter of taking action in good time. In all main line construction projects pipe coating should receive great care. First rate materials, careful application, and inspection are essential. In rocky ground the operations of ditch padding and lowering-in must receive continuous attention. The presence of a layer of fine dirt below and around the pipe, not only protects the coating but facilitates cathodic protection in rocky ground. In arid desert soil one cannot expect completely to arrest the process of corrosion once it has begun. Accordingly cathodic protection should be applied without delay following completion of the pipeline.

Conclusions

1. The effective application of cathodic protection is dependent on favorable conditions to a greater degree than was previously realized.

2. The favorable condition of prime importance is a high-class coating. This should be surrounded by a substantial thickness of selected fine soil.

3. The nature of the ground also is a controlling condition and in a favorable environment poorly coated or even bare steel can be prevented from corroding by cathodic protection.

4. Arid desert soils often constitute an unfavorable environment and only in so far as this disadvantage is offset by a high-class coating can cathodic protection be expected to be effective.

5. In desert soils the successful cathodic protection of an already corroded line is a difficult and uncertain undertaking. The screening effect of corrosion scale makes it difficult both to obtain general polarization and to check that it has, in fact, been obtained.

6. The cathodic protection of lines in moist soil of low conductivity is straightforward and effective.

7. In arid desert soils, because it is a difficult and uncertain matter to arrest corrosion once it has started, cathodic protection should be applied immediately following construction of any line.

Acknowledgments

The author wishes to express his thanks to the Management of the Iraq Petroleum Company, Limited, for permission to publish this paper, and to his colleagues on whose collective experience it is based.

DISCUSSION

Comments by L. P. Sudrabin, Consultant to Electro-Rust-Proofing Corporation, Belleville, New Jersey:

The author has made an excellent appraisal of the special conditions contributing to the severe corrosion experienced on buried pipelines in arid desert soils. A good pipeline coating system, able to withstand the temperature, pressure and voltage stresses, is most essential in obtaining the corrosion control required.

In an engineering study of the corrosion control program on large diameter pipelines in arid desert regions, between

TABLE 2—Leaks on 12-Inch Lines, 665 Miles Total Length

Year	1945	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60
Leaks..	55	44	24	24	53	73	31	109	80	68	31	85	325	104	60	56
									←CATHODIC PROTECTION→							

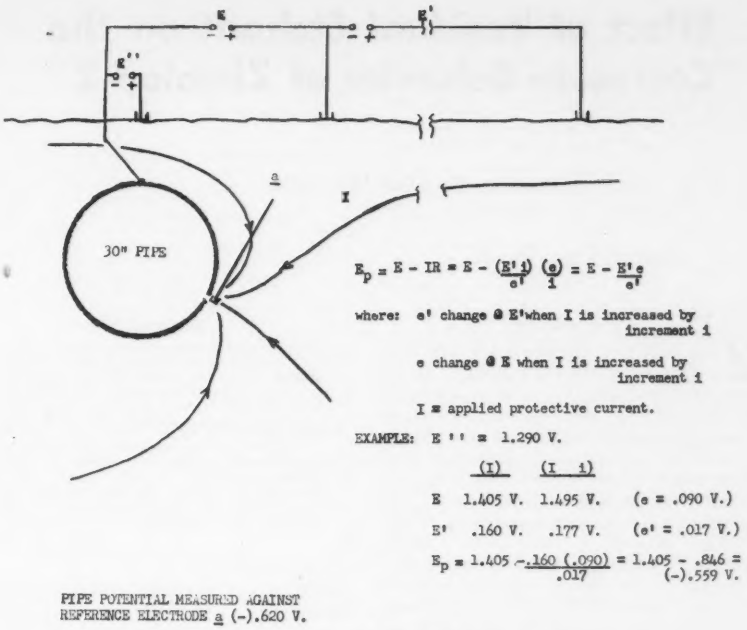
the Persian Gulf and the Mediterranean, the discusser has observed that the pipe potential measured against a reference electrode positioned on the ground surface was not truly indicative of the potential existing at the pipe surface. It was found that a pipe potential of —.85 volt to a $CuSO_4$ half cell positioned on the ground surface, particularly in high "apparent" resistivity soils, does not provide all the corrosion control that can be obtained from cathodic protection.

A representative observation on a poorly coated pipeline in high resistivity desert soil, where severe corrosion has been experienced, is illustrated in Figure 1. An exposed strip, attributable to shoe damage during field coating application, was known to extend longitudinally along a section of the pipeline. The pipe potential measured to a reference electrode positioned over the pipe (E″) and at the flaw (a) was —1.290 volts and —.620 volts, respectively. The pipe potential measured to the probe electrode positioned on the well coated surfaces closely approached —1.290 volts.

An approximation of the pipe potentials to a close reference electrode was determined by a modification of a method for IR drop correction proposed by J. M. Pearson.[1] The random disposition of coating flaws and non-uniform soil resistivities will not provide the symmetrical distribution of potential gradients needed by the Pearson Method. Because earth field and stray current gradients were found to be negligible on the pipeline, two matched CuSO, half cells positioned approximately three feet and 30 feet to one side of the pipeline were used to measure E, E′ and the incremental gradient changes e, e′ attributable to increasing the applied current I by an amount i. In Figure 1, it will be noted that the calculated potential of the pipe (E_p) —.559 volt closely approaches the measured value at (a)—.620 volt.

In Figure 1, most of the IR drop included in the pipe potential measured to a reference electrode positioned on the ground surface cannot be included in the cathodic polarization effect[2] needed to control the local cell short lines of corrosion current flow.

The Trans-Arabian Pipe Line has experienced a marked reduction in the cumulative leak record by reconditioning and recoating the pipeline in aggressive soil areas where poor and nonadherent coatings occur. The recoating work is supplemented by a vigorous cathodic protection program.


E
E'
E''
a
I
30" PIPE
$E_p = E - IR = E - \frac{(E' i)}{e'} \frac{(e)}{i} = E - \frac{E' e}{e'}$
where: e' change @ E' when I is increased by increment i
e change @ E when I is increased by increment i
I = applied protective current.
EXAMPLE: E'' = 1.290 V.
(I) (I i)
E 1.405 V. 1.495 V. (e = .090 V.)
E' .160 V. .177 V. (e' = .017 V.)
$E_p = 1.405 - \frac{.160\ (.090)}{.017} = 1.405 - .846 = (-).559$ V.
PIPE POTENTIAL MEASURED AGAINST REFERENCE ELECTRODE a (-).620 V.

Discussion Figure 1—E_p pipe potential to close electrode.

Question by Tom Watson, Corrosion Service Limited, Toronto, Ontario, Canada:

Are you not sure that the erratic potentials shown were not the effect of varying IR drops between the electrode and the pipe surface in soils and widely varying resistivity?

Reply by W. C. R. Whalley:

Numerous checks show only small differences in such cases between the potential at ground level over the pipe and at pipe depth. Such differences rarely amount to as much as 0.2 v.

Questions by Glenn R. Roberson, Standard Oil Company of Texas, Houston 1, Texas:

1. Is a potential with the electrode on top of the ditch satisfactory or do they have to get closer to the pipe?

2. Was the coating in good condition where they found little difference in the readings?

Replies by W. C. R. Whalley:

1. We found little difference.

2. Yes, in so far as gross defects were not visible.

(1) Pearson, J. M., *Corrosion*, 3, 549, 1947.

(2) Sudrabin, L. P. and Ringer, F. W., *Corrosion*, 13, 351t (1957) May.

Any discussion of this article not published above will appear in June, 1962 issue

Effect of Residual Etchant on the Corrosion Behavior of Zircaloy-2*

By STANLEY KASS

Abstract

The aqueous corrosion behavior of Zircaloy-2 surfaces contaminated with reaction product from the HNO_3-HF-H_2O etchant was studied. Surfaces slightly contaminated with residual etchant exhibit cloudy surfaces and corrode more rapidly than non-contaminated surfaces. The surfaces of coupons severely contaminated exhibit white continuously spalling oxide films in high temperature water. In 750 F steam, however, the corrosion is characterized initially by a spalling corrosion film followed by an adherent corrosion product. 6.3.20, 2.3.6

Introduction

ZIRCALOY-2 SURFACES are prepared for corrosion exposure in high temperature water and steam by chemical etching in an aqueous solution containing 2.5 to 3.5 percent by volume hydrofluoric acid and 39 percent nitric acid at 90 F.[1,2] Experience over the past few years has shown that the surfaces will be corrosion resistant only if stringent control of the etching process is maintained. "Acid-staining," which is caused by incomplete removal of the etching solution followed by the etchant drying upon the surfaces, generally occurs when the process is not under strict control. It has been previously demonstrated that incomplete rinsing or delays in rinsing of sufficient duration which permit the etchant to completely dry upon the Zircaloy surfaces lead to severe corrosion and extremely high weight losses after exposure to high temperature water and steam. It has also been demonstrated that if the transfer of the component from the etchant to the rinse is not made rapidly, the etchant clinging to the surfaces becomes spent and deposits residual reaction product. Subsequent exposure to high temperature water results in slightly cloudy surfaces involving only slight changes in weight gain values.[3]

At present, very limited quantitative data are available describing the aqueous corrosion behavior of Zircaloy-2 surfaces contaminated with residual etching solution. The following experimental work was performed to evaluate the corrosion properties of the surfaces exhibiting varying degrees of residual etchant contamination.

★ Submitted for publication February 26, 1960.

TABLE 1—Schematic Diagram of Experimental Program*

Test Temperature—Code A
750 F—A-1
680 F—A-2
650 F—A-3
600 F—A-4
Etching Bath Temperature—Code B
70 F—B-1
110 F—B-2
150 F—B-3
180 F—B-4
Delay Time to Rinse Solution—Code C
0 min—C-1
1 min—C-2
5 min—C-3
24-72 hr—C-4

* Four code numbers are used to describe each experimental condition, e.g., A-1, B-1, and C-1 represents coupons tested in 750 F steam after etching at 70 F and rinsed immediately.

Experimental

A total of 256 coupons measuring 1 in x 0.5 in x 0.1 in was machined from one section of strip fabricated from an inert atmosphere-melted Zircaloy-2 ingot. The coupons were randomly grouped into 16 groups of 16 coupons each and were etched in the 3.5 percent HF-39 percent HNO_3-balance H_2O solution for sufficient time to remove 2 mils per surface. Four bath temperatures (70, 110, 150, and 180 F) were used. One group of 16 coupons etched at each bath temperature was rapidly transferred from the etchant to the rinsing solution. A second group was permitted to drain one minute before immersion in the rinse, while the third group drained for five minutes prior to rinsing. The fourth group of coupons was

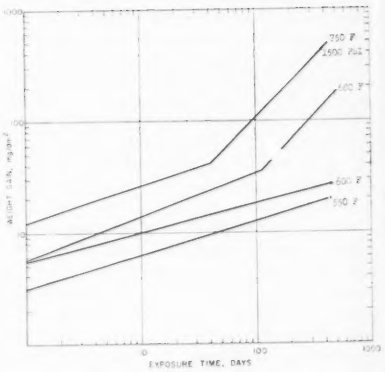


Figure 1—Weight gain-time curves for the corrosion of Zircaloy-2 in high temperature water and steam. 750 F, 680 F and 550 F specimens annealed below 800 C, 600 F specimens as hot rolled.

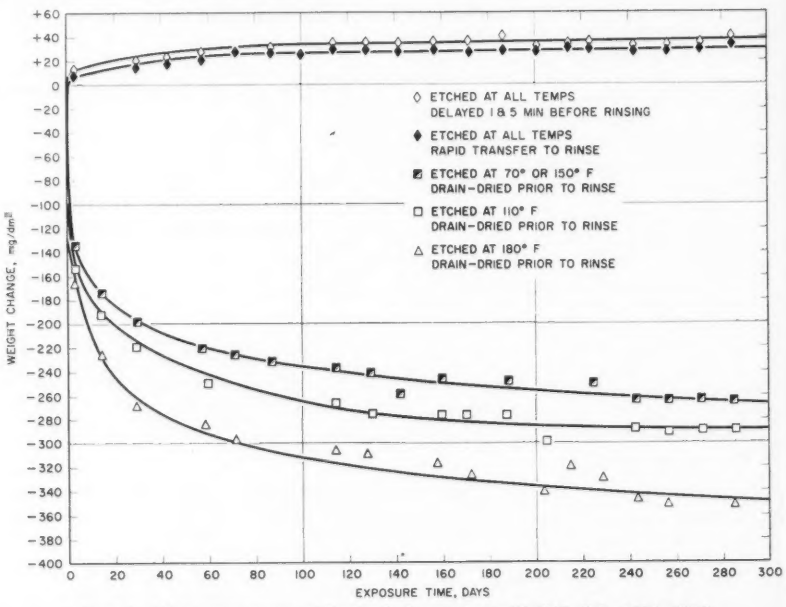

◊ ETCHED AT ALL TEMPS DELAYED 1 & 5 MIN BEFORE RINSING
♦ ETCHED AT ALL TEMPS RAPID TRANSFER TO RINSE
▨ ETCHED AT 70° OR 150° F DRAIN-DRIED PRIOR TO RINSE
□ ETCHED AT 110° F DRAIN-DRIED PRIOR TO RINSE
△ ETCHED AT 180° F DRAIN-DRIED PRIOR TO RINSE
WEIGHT CHANGE, mg/dm²
EXPOSURE TIME, DAYS

Figure 2—600 F water corrosion of Zircaloy-2 after etching in HF-HNO_3-H_2O at 70 to 180 F.

About the Author

STANLEY KASS is with Westinghouse Electric Corporation's Bettis Atomic Power Division, Pittsburgh, Pa. His title is Lead Engineer, Corrosion Section, Nuclear Core Department. Mr. Kass received a BS in chemistry from Ohio University in 1943. His principal field of work is in the corrosion behavior of nuclear reactor materials including zirconium, uranium, hafnium and their alloys.

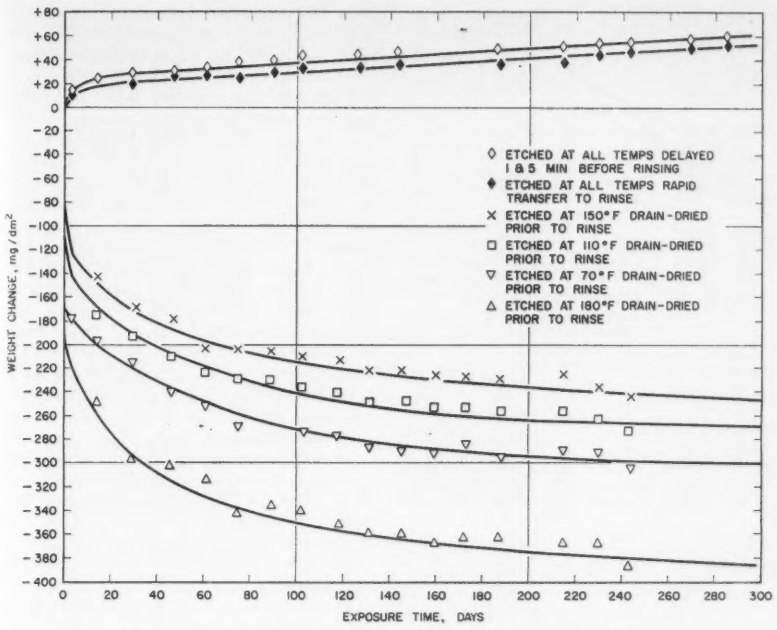

WEIGHT CHANGE, mg/dm²
EXPOSURE TIME, DAYS
◊ ETCHED AT ALL TEMPS DELAYED 1 & 5 MIN BEFORE RINSING
♦ ETCHED AT ALL TEMPS RAPID TRANSFER TO RINSE
× ETCHED AT 150°F DRAIN-DRIED PRIOR TO RINSE
□ ETCHED AT 110°F DRAIN-DRIED PRIOR TO RINSE
▽ ETCHED AT 70°F DRAIN-DRIED PRIOR TO RINSE
△ ETCHED AT 180°F DRAIN-DRIED PRIOR TO RINSE

Figure 3—650 F water corrosion of Zircaloy-2 after etching in HF-HNO_3-H_2O at 70 to 180 F.

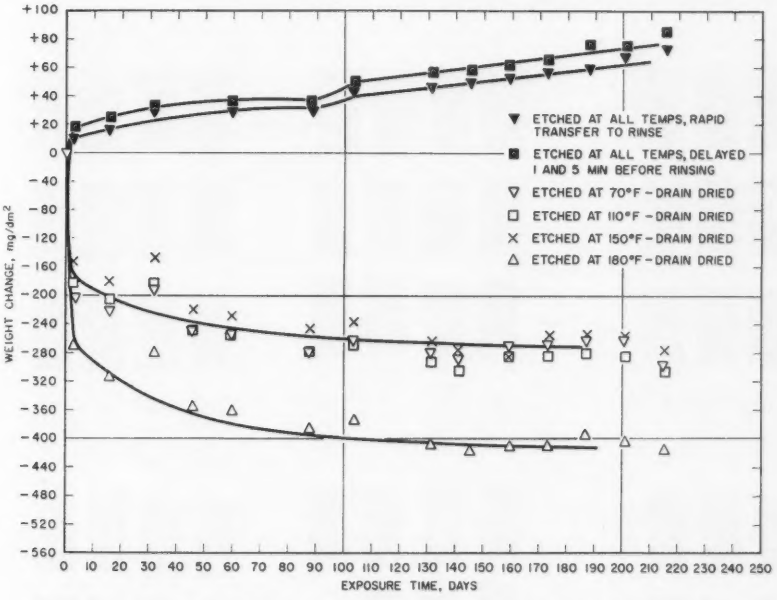

WEIGHT CHANGE, mg/dm²
EXPOSURE TIME, DAYS
▼ ETCHED AT ALL TEMPS, RAPID TRANSFER TO RINSE
■ ETCHED AT ALL TEMPS, DELAYED 1 AND 5 MIN BEFORE RINSING
▽ ETCHED AT 70°F - DRAIN DRIED
□ ETCHED AT 110°F - DRAIN DRIED
× ETCHED AT 150°F - DRAIN DRIED
△ ETCHED AT 180°F - DRAIN DRIED

Figure 4—680 water corrosion behavior of Zircaloy-2 after etching in HF-HNO_3-H_2O at 70 to 180 F.

TABLE 2—Empirical Equations Describing the Corrosion Behavior of Zircaloy-2

Temperature		Medium	Pressure	Equation ΔW in mg/dm², t in Days
550 F	288 C	Water	750 psi	$\log \Delta W = 0.5 + 0.3 \log t$
600 F	316 C	Water	1553 psi	$\log \Delta W = 0.74 + 0.26 \log t$ after 1150 days (ΔW—34) = 0.065 (t—1150)
680 F	360 C	Water	2705 psi	$\log \Delta W = 0.76 + 0.38 \log t$ after 112 days (ΔW—34) = 0.37 (t—112)
750 F	400 C	Steam	1500 psi	$\log \Delta W = 1.1 + 0.32 \log t$ after 41 days (ΔW—41) = 1.27 (t—41)

not rinsed until 24-72 hours had elapsed after pickling. (This condition represents the most severe case of residual etchant contamination.) The coupons in each of the groups were then subdivided into four sections, and corrosion tested in 600, 650, and 680 F water and 750 F steam at 1500 psig using methods previously described.[1] A schematic diagram of the experiment is shown in Table 1.

Results and Discussion

General

The behavior of Zircaloy-2 in high temperature water and steam (Figure 1) has been shown to be characterized by a low corrosion rate followed by a transition from the protective quasi-cubic type kinetics to linear kinetics, the newer corrosion rate being somewhat greater than that prevailing immediately prior to the change.[3,4,5,6] These investigations have shown that the weight gain-time relationship can be expressed by equations of the form

$$\Delta W = kt^n \qquad (1)$$

or its logarithmic form

$$\log \Delta W = \log k + n \log t \qquad (2)$$

where ΔW is the weight gain, t is the time, and k and n are constants for a given temperature. The equations and average constants for the water tests at 550, 600, 680, and the steam tests at 750 F are given in Table 2.

The corrosion behavior of the specimens included in this investigation is represented in terms of weight change during corrosion exposure as a function of time in test for each test temperature. The rate constants in Equation 2 were calculated from the graphically determined slopes. The variations in the corrosion behavior caused by increasing etchant temperatures and increasing delays prior to rinsing are presented in Tables 3, 4, and 5. The experimentally determined weight change values for each of the test groups are depicted graphically as functions of test time in Figures 2, 3, 4, and 5. Photographs of the coupon surfaces after extended exposures at each test temperature and medium are shown in Figures 6, 7, 8, and 9.

Variations in Transfer Time to Rinse

a. **No Delay.** All coupons initially exhibited bright, lustrous oxide films and no evidences of white corrosion product generally associated with accelerated corrosion due to fluoride ion contamination. The corrosion films generally lost the luster as exposure time increased. The corrosion curves and rate constants at all four corrosion test temperatures (Table 3) show excellent agreement with previously reported data (Table 2).

b. **One or five minute delay.** All coupons exhibited patches of white corrosion product overlaying the black zirconium oxide film (Figures 6, 7, 8, 9, and 10). The white corrosion product, which was apparent after only three days exposure at all test temperatures, was adherent and could not be readily removed. Continued corrosion exposure did not alter the surface appearances.

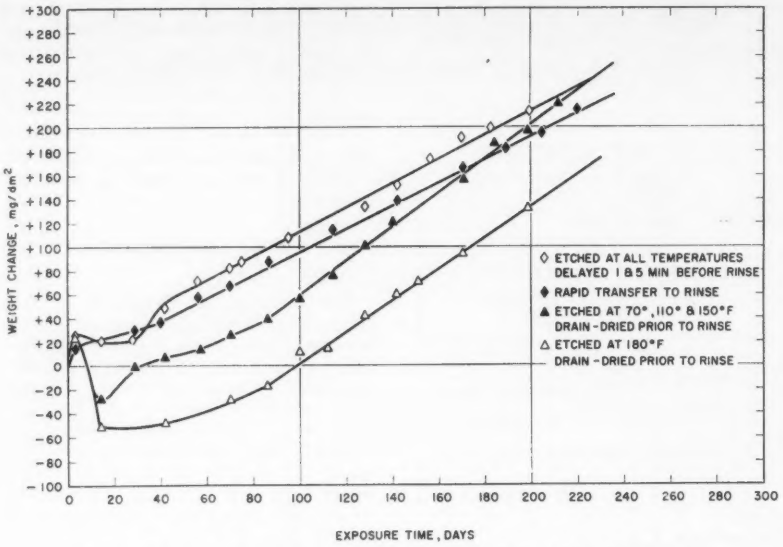

WEIGHT CHANGE, mg/dm²
EXPOSURE TIME, DAYS
◇ ETCHED AT ALL TEMPERATURES DELAYED 1 & 5 MIN BEFORE RINSE
◆ RAPID TRANSFER TO RINSE
▲ ETCHED AT 70°, 110° & 150°F DRAIN-DRIED PRIOR TO RINSE
△ ETCHED AT 180°F DRAIN-DRIED PRIOR TO RINSE

Figure 5—750 steam corrosion of Zircaloy-2 after etching in HF-HNO_3-H_2O at 70 to 180 F.

The corrosion kinetics of the material slightly contaminated with residual etchant is remarkably similar to the uncontaminated material with one notable exception; the contaminated coupons exhibit larger weight gains (thus higher corrosion rates) during the very early stages of corrosion exposure (three days). After the initial exposure period the resultant corrosion rates are identical (Tables 3 and 4).

c. **Twenty-four to seventy-two hour delay.** The corrosion behavior and kinetics in 750 F steam are markedly different from the behavior in water, although all coupons in both water and steam were completely covered with white corrosion product after three days exposure (Figure 10). In 750 F steam, the white corrosion product appeared to be adherent after the 3-day test; however, change in kinetics with associated breakaway corrosion was observed after 14 days exposure. After 28 days exposure, the white oxide films appeared adherent and did not change appearance throughout the test. The corrosion weight change data (Figure 2) verify the visual observations of the change in corrosion kinetics. The linear corrosion rate constant in the post-breakaway region is slightly higher than the post-transition corrosion rate of uncontaminated Zircaloy.

In the aqueous media, white spalling corrosion product was observed after 3 days exposure and spalling was continuously observed throughout the tests. The difference between the thoroughly rinsed or slightly contaminated coupons and the completely acid-stained coupons is quite apparent. While the former exhibited adherent films, the drain-dried coupons exhibited early and continued spalling. The slight variations observed in maximum weight losses in each of the aqueous media tests will be discussed in another section of this report.

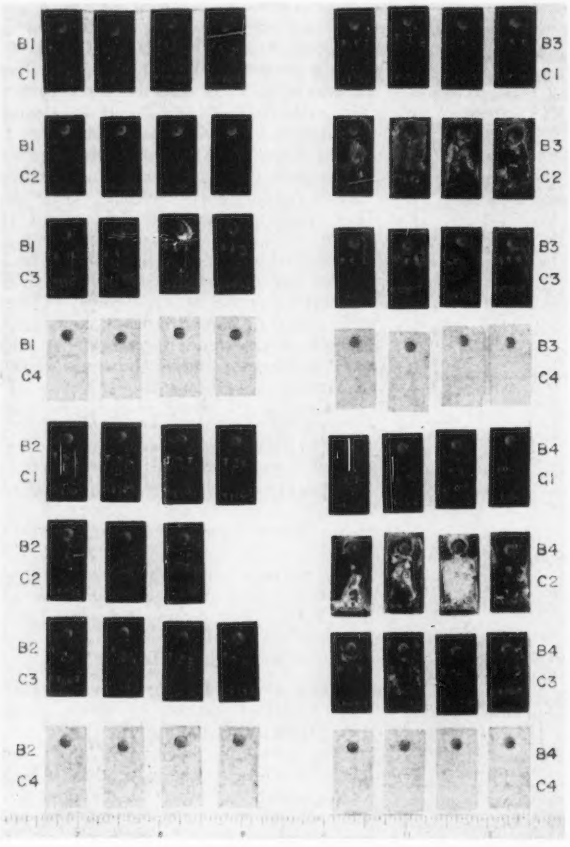


Figure 6—Acid stained Zircaloy-2 corrosion tested for 229 days in 600 F water. Code to samples is found in Table 1.

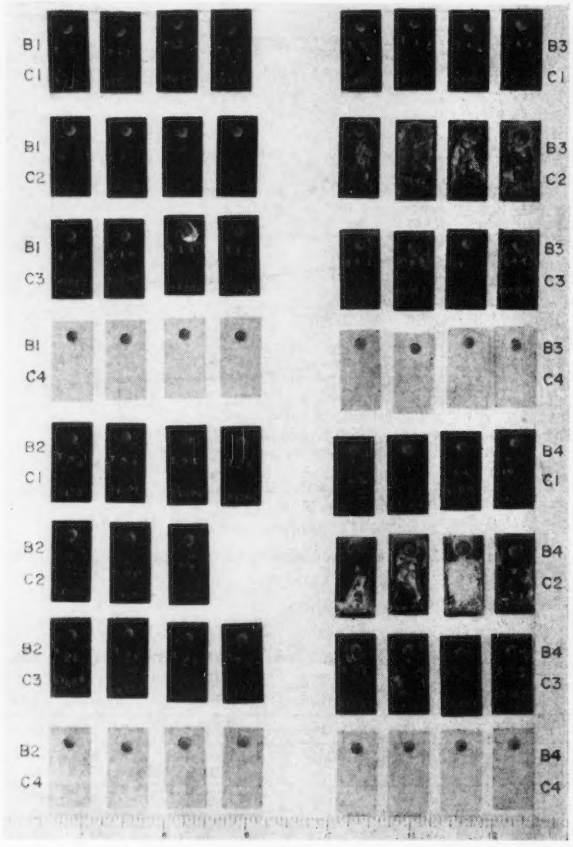


Figure 7—Acid stained Zircaloy-2 corrosion tested for 229 days in 650 F water. Code to samples is found in Table 1.

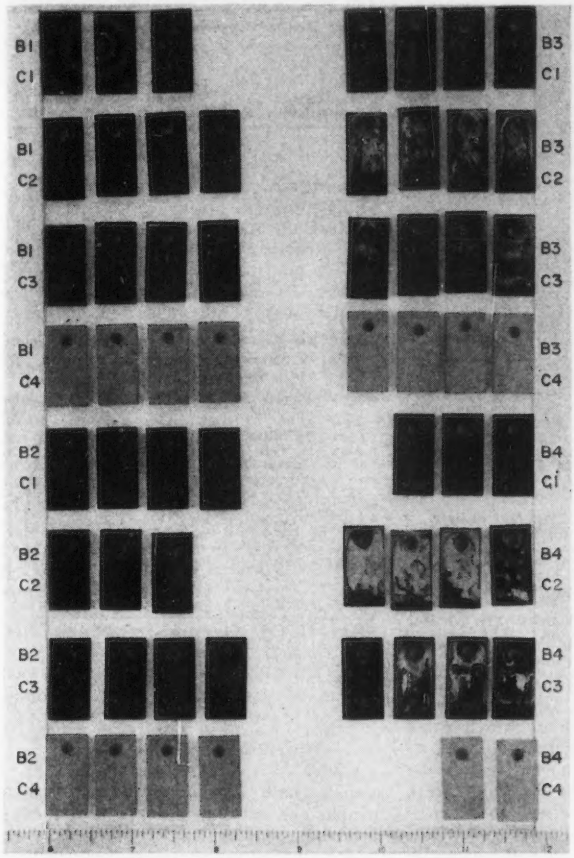


Figure 8—Acid stained Zircaloy-2 corrosion tested for 202 days in 680 F water. Code to samples is found in Table 1.

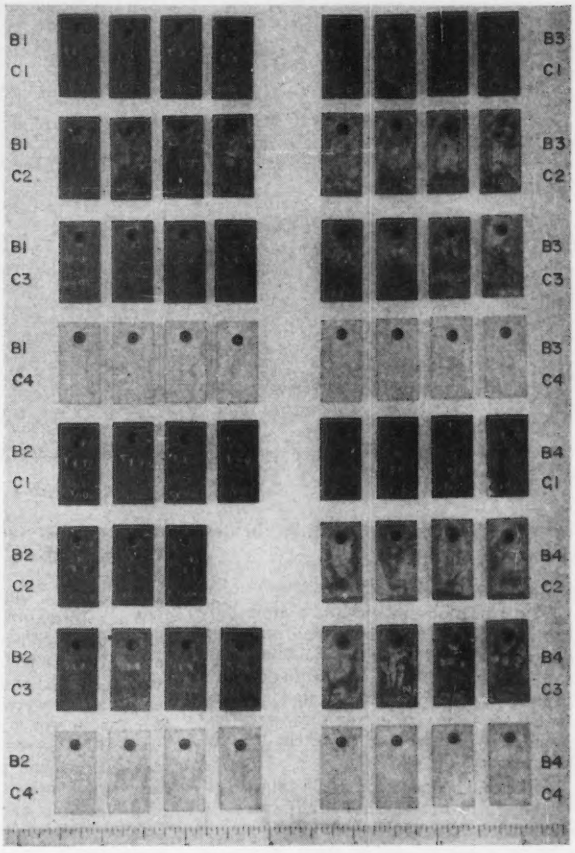


Figure 9—Acid stained Zircaloy-2 corrosion tested for 212 days in 750 F steam. Code to samples is found in Table 1.

TABLE 3—Kinetics of Corrosion of Zircaloy-2 Contaminated with Residual Etchant

Temperature °F	Temperature °C	Test Media	Transfer Time To Rinse	Equation ΔW in mg/dm², Time in Days
600	316	Water	Approx. 5 sec	$\log \Delta W = 0.98 + 0.29 \log t$
600	316	Water	1 or 5 min	$\log \Delta W = 0.93 + 0.33 \log t$
650	343	Water	Approx. 5 sec	$\log \Delta W = 1.0 + 0.28 \log t$
650	343	Water	1 or 5 min	$\log \Delta W = 1.29 + 0.23 \log t$
680	360	Water	Approx. 5 sec	$\log \Delta W = 0.75 + 0.23 \log t$ post transition (Δ W—40) = 0.23 (t—105)
680	360	Water	1 or 5 min	$\log W = 0.96 + 0.28 \log t$ post transition (Δ W—50) = 0.24 (t—105)
750	400	Steam	Approx. 5 sec	$\log W = 1 + 0.3 \log t$ post transition (Δ W—38) = 1.16 (t—40)
750	400	Steam	1 or 5 min	post transition (Δ W—50) = 1.1 (t—40)

Variations in Corrosion Temperature

a. **One or five minute delay.** The dependency of the aqueous corrosion behavior of slightly contaminated Zircaloy-2 surfaces with temperature is seen to follow the general trends noted for Zircaloy-2 properly prepared for corrosion exposure; for example, the corrosion rate increases with increasing corrosion test temperature. The sensitivity of the corrosion test as an inspection tool for "acid-stained" surfaces is markedly clear. Three days exposure at all test temperatures dramatically detected the deleterious effects of the residual etchant.

The data further show a remarkable similarity in the influence of the residual etchant on the corrosion behavior in all three water tests. The mean weight change increases at all temperatures due to "acid-stain" is approximately 8 mg/dm² indicating a contaminated layer of uniform thickness which corroded rapidly during the first few days of exposure. The corrosion kinetics were then normal for the respective temperatures. Thus it is apparent that the residual etchant contamination does not lead to progressive accelerated corrosion and the corrosion properties of the underlying metal are not influenced by the surface contamination.

Similar corrosion behavior is noted in 750 F steam except for transient effects which were observed during the early stages of corrosion. After 40 days exposure in 750 F steam, the corrosion of the residual etchant contaminated material closely parallels the corrosion of material not contaminated with residual etchant.

b. **Twenty-four to seventy-two hour delay.** Specimens severely contaminated with etchant reaction product exhibit corrosion rates in water which increase as the corrosion test temperature is increased. Furthermore, as the kinetics follow the exponential rate equation (Table 5) the corrosion rate at any one temperature decreases with increasing exposure time. Thus, although the corrosion appears progressive and oxide sloughing was observed throughout the tests, maximum metal loss was noted during the initial two weeks exposure.

The 750 F steam tests show different behavior which has been discussed previously. However, it should be pointed out that the maximum metal weight loss in 750 F steam is only 10 percent of the value observed in the water tests. These differences may be explained by the erosive action of the moving water caused

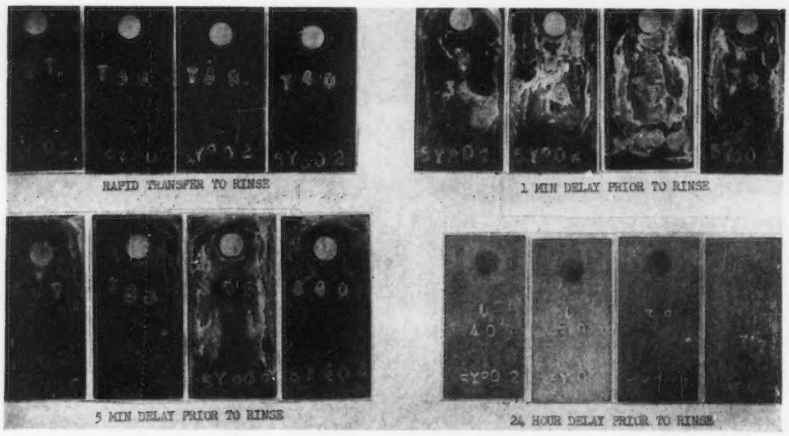


Figure 10—Typical coupon surfaces after 3 days exposure in 650 F water.

by thermal gradients within the test autoclave.

Variations in Etchant Temperature

The variations in etchant temperature are observed to have produced no effects when the transfer of the coupons from the etchant to the rinse was made either rapidly or slowly. The weight change values in each test group show excellent agreement regardless of etchant temperature employed.

Different behavior of the "drain-dried" coupons are noted. In 600 F water the coupons etched at 70 and 150 F exhibit similar behavior, while those etched at 110 and 180 F corroded more rapidly. In 650 F water, dissimilar weight losses are noted for each etching temperature. These differences do not show trends dependent upon the etchant temperature and are believed to have been caused by test techniques. After the coupons "drain-dried" for 24 to 72 hours, they were rinsed by immersion in flowing tap water, rinsed in static deionized water, wiped with ethyl alcohol and air dried. No specific attempts were made to remove all residual etchant product from the surfaces. The coupons were then weighed and submitted to corrosion test. Furthermore, the coupons were processed in etchant temperature groups, for example, all coupons for the 70 F etchant series at one time, the coupons for the 110 F etchant series at another time, etc. The techniques resulted in variations in the residual etchant product remaining on the surfaces prior to corrosion exposure and the coupons which contained greater amounts of residual etchant corroded more severely.

Conclusions

When Zircaloy-2 surfaces are rinsed immediately and thoroughly after etching in a nitric acid-hydrofluoric acid bath, subsequent corrosion exposure in high temperature water and steam produces black adherent oxide films which grow slowly with increasing exposure. The corrosion resistance of properly rinsed surfaces is not influenced by the temperature of the etching bath.

Zircaloy-2 surfaces slightly contaminated with residual etchant generally exhibit cloudy surfaces or areas of adherent white corrosion product overlaying black oxide films after aqueous corrosion exposure. The behavior is generally characterized by accelerated corrosion for a short period followed by a rate equal to that of properly prepared surfaces. The thickness of the contaminated layer is not influenced by etching bath temperature or variations from 1 to 5 min delay in transfer from the etchant to the rinse. These layer thicknesses reach a uniform value invarient with test temperature over the range 600 to 680 F.

The surfaces of coupons severely contaminated with residual etchant exhibit white spalling oxide films after exposure to high temperature water and steam. In 750 F steam, the corrosion, after an initial period of spalling, proceeds at a rate slightly higher than uncontaminated surfaces and forms adherent oxide films. The corrosion rate is not influenced by etching bath temperature. In water media, the surfaces exhibit white, continuously spalling oxide films; the corrosion is progressive, follows exponential rate laws at each test temperature and increases with increasing corrosion test temperature.

TABLE 4—Post Transition Corrosion Rates of Etchant Contaminated Zircaloy-2

Test Temperature, °F	Transfer Time to Rinse	Linear Corrosion Rate mg/dm²-Day
680	Approx. 5 sec	0.23
680	1 or 5 min	0.24
750	Approx. 5 sec	1.16
750	1 or 5 min	1.09
750	24 to 72 hr	1.25
		1.40

TABLE 5—Kinetics of Corrosion of Zircaloy-2 Severely Contaminated with Residual Etchant

Test Temperature	Etchant Temperature	ΔW = Weight Loss, mg/dm² Equation-t = Time, Days
600 F	70 F or 150 F	$\log \Delta W = 2.06 + 0.15 \log t$
	110 F	$\log \Delta W = 2.11 + 0.15 \log t$
	180 F	$\log \Delta W = 2.16 + 0.17 \log t$
650 F	70 F	$\log \Delta W = 2.17 + 0.13 \log t$
	110 F	$\log \Delta W = 2.07 + 0.15 \log t$
	150 F	$\log \Delta W = 1.99 + 0.16 \log t$
	180 F	$\log \Delta W = 2.27 + 0.13 \log t$
680 F	70 F or 110 F or 150 F	$\log \Delta W = 2.07 + 0.17 \log t$
	180 F	$\log \Delta W = 2.35 + 0.12 \log t$

Acknowledgement

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Corrosion Inhibitors for Hot-Carbonate Systems*

By D. BIENSTOCK and J. H. FIELD

Abstract

The evaluation of the effectiveness of various inhibitors on the corrosion of steel in boiling solutions of potassium carbonate saturated with carbon dioxide and hydrogen sulfide is cited. The corrosion of carbon steel in a carbonate solution saturated with carbon dioxide can be eliminated with a 0.2 percent concentration of either potassium chromate or sodium metavanadate. In solutions of carbonate saturated with carbon dioxide and hydrogen sulfide no corrosion is obtained with 0.2 percent sodium metavanadate or 0.15 percent vanadium pentoxide. The alkaline-sulfide mixture destroys oxidizing inhibitors such as potassium chromate. N-alkyl trimethylene diamines at a concentration of 0.1 percent reduce the corrosion rate to about 5 mpy. Sodium metasilicate, at a concentration of 0.6 percent, completely inhibits the corrosion, but deposits free silica in the system. Commercially available cladding materials such as the epoxy-coatings also prevent corrosion. 5.8.2, 6.2.3

Introduction

PROBLEMS OF corrosion became evident early in the development of the hot-carbonate process by the Federal Bureau of Mines.[1,2] Hot solutions of potassium carbonate for absorbing carbon dioxide are highly corrosive to carbon steel. The corrosion rate in a 40-percent solution saturated with carbon dioxide is 340 mpy. Stainless steels, such as Types 304 and 347, suffer negligible attack[3] but their use for all equipment would be too costly.

Corrosion is greatly reduced when hydrogen sulfide and carbon dioxide are simultaneously absorbed. A 40-percent solution of potassium carbonate saturated with carbon dioxide and 0.3 to 10 percent hydrogen sulfide corroded steel at the rate of 10 mpy, a reduction of 96 percent. Because operating difficulties occur even at this mild corrosion rate, inhibitors are desirable. A laboratory investigation on the use of inhibitors for the K_2CO_3-CO_2 and the K_2CO_3-CO_2-H_2S system was undertaken, and this report discloses the results.

Experimental Procedure

The experimental procedure has been reported in detail previously.[3] Figures 1 and 2 show a schematic diagram of the corrosion testing unit and corrosion vessel, respectively.

Discussion

Use of Inhibitors in the K_2CO_3-CO_2 System

A variety of reagents, inorganic and organic, were tested as possible inhibitors for the highly corrosive system of potassium carbonate saturated with carbon dioxide. The results of some of these tests are shown in Table 1. Difficultly soluble reagents were heated for 24 hours in the boiling carbonate solution in the absence of carbon dioxide in an attempt to develop a protective coating on the steel surface. At the end of this period carbon dioxide was introduced and the timing of the test began.

Potassium Chromate

Potassium chromate is very effective as an inhibitor. In the test apparatus at a concentration of 0.2 percent, corrosion of the steel was eliminated in a 40- and 50-percent solution of potassium carbonate saturated with carbon dioxide. The steel disks at the end of a 30-day test were as shiny and unchanged in appearance as at the start. The effectiveness of potassium chromate as an inhibitor was subsequently confirmed in pilot plant tests. Its ability as an inhibitor for galvanic corrosion as a result of contact of dissimilar metals, was also studied. Stainless steel disks of the 304, 347, and 410 series were fastened face to face with disks of mild carbon steel, giving a unity area ratio of the disks. The rate of corrosion of the couples in a 40-percent carbonate solution saturated with carbon dioxide containing 0.2 percent potassium chromate was determined. These results are shown in Table 2. Almost complete protection from corrosion was obtained for the stainless steel-carbon steel couples under the conditions of these tests. This degree of inhibition is significant for a commercial plant where stainless and mild steel fittings are coupled together.

In the early pilot plant development of the hot-carbonate process by the Bureau of Mines, severe corrosion of the carbon steel equipment occurred when an uninhibited solution of potassium carbonate was used to absorb carbon dioxide. However, when potassium chromate was added to the carbonate solution and maintained at a concentration of 0.2 percent, corrosion was halted. It should be stressed that when newly installed equipment is used for the first time, the potassium carbonate solution containing the chromate should be circulated before introducing the carbon dioxide-containing gas. This procedure will precoat the fresh metallic surfaces. During the first week of actual operation the concentration of chromate should be carefully watched and replenished to maintain a value of 0.2 weight percent. Thereafter the decrease in concentration of inhibitor is slight. For commercial operation the less expensive dichromate may be substituted for chromate. In the alkaline solution the dichromate ion is converted to the chromate.

Pitting may be encountered occasionally as a result of crevice corrosion under scale or other deposits or under overlapping metal. These potential sources of trouble can be eliminated by proper design of equipment. There is still some slight difficulty existing in some areas which may be described as a combined corrosion-erosion problem. The difficulty occurs in such places as the pressure let-down of the spent solution when carbon dioxide and steam are flashed, and to a lesser degree in places of high linear velocity or sharp change in direction. These difficulties can also be avoided by proper design or by substituting stainless for carbon steel at the trouble spots. The carbon-stainless steel couple produced does not give any difficulty in this system. If temperatures of the order of 150 C are to be employed in the reboiler tubes for regeneration, the corrosion is accelerated and use of stainless tubes is recommended.

Sodium Metavanadate

Sodium metavanadate in concentrations of 0.1 to 0.2 weight percent eliminated corrosion of the steel disks in a 40-percent carbonate solution saturated with carbon dioxide. The result of a 30-day test is shown in Table 1. The concentration of vanadate remained constant throughout the test.

Potassium Nitrite

The corrosion of mild steel was erratic when potassium nitrite was used as inhibitor. At a concentration of 0.3 percent potassium nitrite, some of the steel disks

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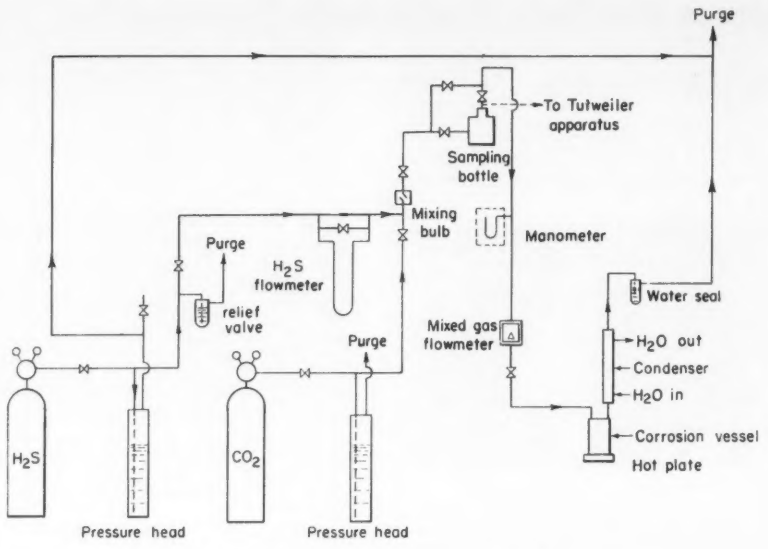

Figure 1—Scematic flow diagram of corrosion-testing apparatus.

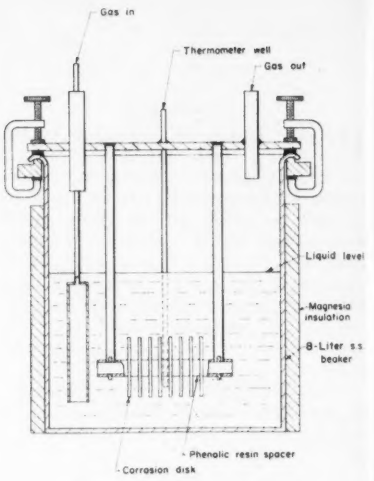

Figure 2—Corrosion test vessel.

TABLE 1—Tests of Possible Inhibitors in the Corrosion of Carbon Steel in Boiling 40-percent Solutions of Potassium Carbonate Saturated with Carbon Dioxide

Inhibitor	Concentration of Inhibitor[a], Weight Percent	Length of Test, Days	Corrosion Rate, mpy
Potassium chromate	0.2	30	<0.1
Sodium metavanadate	.2	30	<0.1
Thiourea	.5	13	46[b]
N-Alkyl trimethylene diamine	.5	30	160[b]
Potassium sulfate	.2	14	170
Potassium nitrite	.3	15	[c]
Uninhibited	..	14	340

[a] Concentration at start of test.
[b] Disks pretreated 24 hours with inhibitor in boiling carbonate solution in absence of carbon dioxide.
[c] Three disks <1 mpy, 2 disks 240 mpy, 1 disk completely corroded.

TABLE 2—Corrosion Rates of Stainless Steel-Carbon Steel Galvanic Couples in a 40-percent Solution of Potassium Carbonate Saturated with Carbon Dioxide and Inhibited with 0.2 Percent Potassium Chromate

Temperature of Boiling Solution: 109C

Galvanic Couple	Length of Test, Days	Corrosion Rate, mpy
410 stainless-carbon steel	30	0.14, 410 stainless steel .01, carbon steel
347 stainless-carbon steel	30	.02, 347 stainless steel .03, carbon steel
304 stainless-carbon steel	30	.01, 304 stainless steel .03, carbon steel

TABLE 3—Tests of Possible Inhibitors in Corrosion of Carbon Steel in Boiling Solutions of Potassium Carbonate Saturated with Carbon Dioxide and Hydrogen Sulfide (99.7/0.3)

Inhibitor	Concentration of Inhibitor[a], Weight Percent	Length Test, Days	Corrosion Rate, mpy
Ammonium thiocyanate	0.5	14	19
Diphenylthiourea	.5	14	19[b]
N-alkyl trimethylene diamine (coconut base)	.5	29	9[b]
N-alkyl trimethylene diamine (tallow base)	.02	29	5[b]
N-alkyl trimethylene diamine (tallow base)	.1	29	7[b]
Mercaptobenzothiazole	.5	15	25
Potassium chromate	.2	91	8
Potassium ferrocyanide	.5	14	9
Potassium ferricyanide	.5	15	12
Potassium nitrite	.5	30	70[c]
Potassium phosphate	.5	14	16
Potassium silicate	3 SiO_2	42	8
Sodium metaphosphate	.5	14	16
Sodium metasilicate	←—See Table 4—→		
Sodium metavanadate	.2	30	<0.1
Sodium molybdate	.5	14	18
Tannic acid	.2	14	12
Thiourea	.5	13	15[b]
Trisodium phosphate	.5	14	16
Vanadium pentoxide	.15	30	<0.1
Uninhibited	.....	30	12

[a] Concentration at start of test.
[b] Disks pretreated 24 hours with inhibitor in boiling carbonate solution in absence of acid gases.
[c] Poor reproducibility characteristic of this system.

corroded and the products of corrosion deposited as a film on the remaining disks. When the potassium nitrite concentration was increased to 0.5 percent in another test, one disk completely corroded while the 5 remaining disks all gained weight. During the period in which the steel was corroding, potassium nitrite was added to maintain the original concentration. However, after the disk dissolved, the concentration of the potassium nitrite remained constant for the duration of the test.

Use of Inhibitors in the K_2CO_3-CO_2-H_2S System

Although the corrosion in the K_2CO_3-CO_2-H_2S system is considerably less than in the K_2CO_3-CO_2 system and can be tolerated in steel equipment, it would be preferable from an operating standpoint to inhibit the corrosion completely. Corrosion of steel even at reduced rates results in sludges being formed, strainers clogged, efficiencies of heat transfer across fouled surfaces reduced, and periodic replacement of corroded valves, pump impellers, etc. Several of the more common inhibitors were tested, and the results of the tests are shown in Table 3.

Sodium Metavanadate

Sodium metavanadate at a concentration of 0.2 weight percent inhibited the corrosion of mild steel in a 40-percent carbonate solution saturated with a mixture of carbon dioxide and hydrogen sulfide (99.7 to 0.3 volume ratio). As shown in Table 3, the corrosion rate in a 30-day test was less than 0.1 mpy. Chemical analysis showed that the concentration of vanadate remained constant throughout this test. In other tests a slight loss in

vanadate occurred, and periodic replenishment of the sodium metavanadate was necessary to maintain the initial concentration. However, no corrosion occurred in any of the tests. In tests in which the vanadate concentration was allowed to decrease to less than 0.05 percent, corrosion protection was no better than that obtained with hydrogen sulfide in a noninhibited solution.

Vanadium pentoxide acted similarly to sodium metavanadate as a corrosion inhibitor. As vanadium pentoxide is cheaper than the sodium vanadate, the pentoxide would be the preferred form for commercial use.

Potassium Chromate

When hydrogen sulfide is absorbed by the potassium carbonate, a reducing solution forms. The chromate ion is quantitatively reduced to chromic oxide and precipitates from the solution, and the corrosion-inhibiting effect of potassium chromate is destroyed.

At the end of a 91-day test the rate of corrosion of carbon steel in a 40-percent solution of potassium carbonate saturated with carbon dioxide and hydrogen sulfide (97 to 3 volume ratio) and containing initially 0.2 percent potassium chromate was 8 mpy. A 31-day test gave a value of 9 mpy. These values are equivalent to the rate in a noninhibited solution at the end of 30 days. However, a test of 7 days with potassium chromate indicated a corrosion rate of <0.1 mpy. Evidently sufficient chromate was still present to account for this inhibition. The results with potassium chromate emphasize the need for extended corrosion tests, since a test of short duration may give erroneous data.

N-Alkyl Tremethylene Diamines

The N-alkyl tremethylene diamines offer some protection. As indicated in Table 3 corrosion rates ranged from 5 to 9 mpy in the presence of carbon dioxide and hydrogen sulfide in a volume ratio of 99.7 to 0.3 for a 29-day test. This rate may be compared with 12 mpy in an uninhibited solution. Unfortunately these oil-soluble fatty diamines do not protect the steel in the absence of hydrogen sulfide and thus would be limited to the scrubbing of gas containing some hydrogen sulfide.

Sodium Metasilicate

The neutral salt, sodium metasilicate, showed some promise in the laboratory corrosion tests. A concentration of 1 percent of sodium metasilicate forms a heavy, uniform, tenacious coating on the steel, protecting it from corrosion. However, at this concentration of metasilicate, deposition of free silica occurs in the solution. This deposition would inevitably lead to operative difficulties in a commercial unit. At lower concentrations of the metasilicate, such as 0.4 to 0.5 weight percent, deposition of silica is much less, although still not low enough for successful operation of scrubbers. The corrosion rate, as shown in Table 4, for concentrations of 0.4 to 0.5 percent metasilicate is less than in an uninhibited solution for the first 2 months of the test, but then increases. Although not shown in the table, increasing the silicate concentration has no retarding effect once corrosion starts. By pretreating the steel with an aqueous solution containing 1 percent sodium metasilicate for 48 hours, corrosion can be eliminated for extended periods by maintaining a concentration of only 0.6 percent sodium metasilicate. However, even at this concentration of metasilicate, deposition of silica still occurs which would render impractical its use in absorption equipment. Longer periods of pretreatment did not reduce the concentration of the metasilicate required to inhibit the corrosion.

Potassium Nitrite

The nitrite ion is completely reduced to ammonia as the carbonate solution absorbs hydrogen sulfide. The ammonia that evolved in the presence of carbon dioxide was deposited in the condenser as ammonium carbonate. Poor reproducibility of the tests and accelerated corrosion of some of the disks occurred.

TABLE 4—Corrosion Rates of Carbon Steel in a Boiling 40-percent Solution of Potassium Carbonate Saturated with Carbon Dioxide and Hydrogen Sulfide in a Ratio of 97 to 3 Using Sodium Metasilicate as Inhibitor

TEMPERATURE OF BOILING SOLUTION: 109 C

Concentration of Na_2SiO_3, Weight Percent	No Pretreatment	24-Hr.[a] Pretreatment	48-Hr.[a] Pretreatment	7-Day[a] Pretreatment
1.1	Run 57, 30 days—nil[b] 60 days—nil[c] 88 days—nil			
0.9		Run 87, 21 days—nil 42 days—nil 64 days—nil		
0.8	Run 84, 21 days—10 mpy		Run 91, 21 days—nil 42 days—nil 62 days—nil	
0.6	Run 79, 29 days—9 mpy	Run 86, 21 days— <.1 mpy 42 days— .7 mpy 63 days— .7 mpy	Run 90, 21 days— <.1 mpy 42 days—nil 62 days—nil	
0.5	Run 77, 32 days—3 mpy 60 days—5 mpy 90 days—8 mpy			
0.4	Run 73, 21 days— <.1 mpy 42 days—3 mpy 63 days—7 mpy	Run 74, 21 days— 1 mpy 42 days— 7 mpy 63 days—12 mpy		Run 27, 21 days—6 mpy
0.2	Run 42, 14 days—14 mpy	Run 68, 21 days—9 mpy		Run 96, 29 days—10 mpy

[a] "Pretreatment"—heating disks in boiling aqueous solution containing 1 percent Na_2SiO_3.
[b] "Nil"—no corrosion, disk actually gained in weight.
[c] Steel, removed at end of 60 days with a heavy uniform coating, corroded when placed in a new solution without inhibitor.

Use of Cladding Materials

Steel surfaces in the absence of inhibitors may be protected with commercial coating such as the phenolic or coal tar epoxies. These coatings, as shown in Table 5, hold up well in boiling solutions of carbonate saturated with carbon dioxide and hydrogen sulfide, and prevent attack of the metal beneath. To be most effective, the coating materials have to be applied to the steel equipment with care particularly at edges. The coating, if not reinforced with additional coating at such areas, may separate and peel, permitting accelerated anodic corrosion at these small bare spots. No study was made of the effects of erosion of a flowing carbonate solution and of thermal expansion and contraction upon the tenacity of these coatings.

Conclusions

Various inorganic reagents were tested as potential corrosion inhibitors in boiling solutions of potassium carbonate saturated with certain acid gases. Potassium chromate at 0.2 percent is very effective—completely inhibiting the corrosion of mild carbon steel and even galvanic couples of stainless and carbon steel in carbonate solutions saturated with carbon dioxide. Tests using chromate inhibitor in the pilot plant confirmed the laboratory results. In the presence of hydrogen sulfide either with or without carbon dioxide, the chromate ion is reduced, destroying its effectiveness as an inhibitor.

Although the corrosion rate of a boiling solution of carbonate saturated with carbon dioxide and hydrogen sulfide, obtained in laboratory tests, would be acceptable in carbon steel equipment of satisfactory design, complete protection is obtained with sodium metavanadate and vanadium pentoxide as inhibitors. Carbon steel disks in tests of 30-day duration with a boiling 40-percent carbonate solution saturated with carbon dioxide and hydrogen sulfide (99.7-0.3 volume ratio) and containing 0.2 percent sodium metavanadate or 0.15 percent vanadium pentoxide showed no significant corrosion. Since the less expensive vanadium pentoxide prevents corrosion of steel in carbonate solutions in presence of carbon dioxide or in mixtures of carbon dioxide and hydrogen sulfide, use of this inhibitor is therefore recommended when: (1) both acid gases are present, or when (2) carbon dioxide is scrubbed for

TABLE 5—Resistance of Cladding Materials to Boiling, 40-percent Solution of Potassium Carbonate Saturated with Carbon Dioxide and Hydrogen Sulfide

Cladding Material	Form Used	Ratio CO_2 to H_2S	Length of Test, Days	Remarks
Phenolic Epoxy, Company A.	Steel coated by supplier, 10 mils thick.	97 to 3 90 to 10	19 29 48, total	Coating unaffected.
Phenolic Epoxy, Company B.	Steel hand dipped	99.7 to 0.3	30	Small patches of coating flaked off; the major portion of the coating remained intact and unaffected.
Coal Tar Epoxy, Company C.	Steel coated by supplier	99.7 to 0.3 90 to 10	30 29 59, total	Coating unaffected.
Portland Cement, Type 1....	Cube[a], 2 in.	97 to 3	30	Edges intact, one face roughened.

[a] Mortar mixed and molded according to ASTM C 109, and vibrated for 5 minutes.

a period followed by a mixture of both carbon dioxide and hydrogen sulfide. When carbon dioxide is present and hydrogen sulfide is never expected, the less expensive sodium dichromate would be preferred for plant use.

Complete protection can also be obtained against solutions saturated with a mixture of carbon dioxide and hydrogen sulfide by the use of sodium metasilicate at a concentration of 0.6 percent. Protection with the latter reagent occurs through the formation of an inert, dense, and tenacious deposit on the steel; metasilicate inhibition, however, introduces an operational problem by depositing free silica in the solution under these conditions and consequently is not recommended.

The N-alkyl trimethylene diamines at 0.02 to 0.5 weight percent give moderate protection, reducing the corrosion rate from 12 to 5-9 mpy. These compounds are effective only when hydrogen sulfide is present.

Epoxy cladding materials prevented corrosion and held up satisfactorily in the laboratory corrosion tests with boiling solutions of potassium carbonate saturated with carbon dioxide and hydrogen sulfide. Although tests were conducted only with mixed acid gases, the nature of the protection should make it effective against either gas alone. The effectiveness of these films in a commercial unit would depend on their ability to withstand stresses of fluid flow and thermal changes.

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Any discussion of this article not published above will appear in June, 1962 issue

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Authors of technical papers on corrosion are invited to submit them for review without invitation to the Editor of Corrosion. Write for "Guide for the Preparation and Presentation of Papers" sent free on request to prospective authors.

Reaction of Zirconium With Carbon Dioxide at 550-750 C*

By S. J. GREGG,[1] R. J. HUSSEY[2] and W. B. JEPSON[3]

Abstract

The kinetics of the oxidation of zirconium in Carbon-14 labelled carbon dioxide have been investigated at 550, 650 and 750 C with the vacuum microbalance. The amount of carbon deposited on the sample surface during oxidation in carbon dioxide was only about 2 percent of the total weight gain at each of the temperatures. In carbon dioxide, the rate of oxidation first decreased and then increased with time indicating breakaway. In runs in oxygen (carried out for comparative purposes), the rate continuously decreased with time and breakaway did not occur within the period of the experiments. Maximum weight gain was 900 $\mu g/cm^2$.

6.3.20, 3.4.6

Introduction

WHILE THE OXIDATION of zirconium in oxygen and in air has been extensively studied,[1-5] work on the oxidation in carbon dioxide is more limited.[6-8] Hayes, Roberson and Robertson[7] measured the weight gain of specimens after 1 and 24 hours oxidation at a number of temperatures between 600 and 1000 C; the detailed course of the oxidation was not investigated, and the deposition of carbon was inferred from the appearance of the oxidized samples. O'Driscoll, Tyzack and Raine[8] studied the oxidation of zirconium and a number of zirconium alloys in carbon dioxide at pressures between 1 and 8 atm and at temperatures in the range 400-800 C.

The present work constitutes a brief investigation of the over-all kinetics of the oxidation of zirconium in carbon dioxide at 550, 650 and 750 C in the 'thin film' region of oxidation. By isotopically labelling the carbon dioxide with carbon-14, it was possible at the end of each run to estimate the amount of carbon deposited on the sample. The course of the oxidation in oxygen was also studied in order to provide comparative data.

Experimental

The zirconium was obtained in the form of 1/32-inch sheet.* The quoted analysis is given in Table 1. For each run, a sample measuring 2.5 cm x 1 cm was cut from the sheet and was abraded first on Grade 0 and then on Grade 00 emery under petroleum ether, before being degreased in a benzene still.

The course of the oxidation was followed by measuring the weight gain of the sample on a vacuum microbalance[9,10] sensitive to 5 μg; the pressure of the gas, whether carbon dioxide or oxygen, was 10 cm.

At the start of a run, the sample was first outgassed at room temperature to a pressure of 10^{-6} mm. The furnace was then switched on, and outgassing continued as the temperature rose; after the sample had been held at run temperature for 75 min, the oxidizing gas was admitted to the balance case and readings taken at suitable intervals. The furnace was controlled to $\pm$ 0.25 C with a Sunvic RT2 controller. The temperature was measured with a calibrated chromel-alumel thermocouple.

The radioactive carbon dioxide was prepared[11] by heating a mixture of lead chloride and barium carbonate to which had been added C^{14} in the form of barium carbonate; the evolved gas was dried by distilling through a column packed with phosphorus pentoxide, and traces of air were removed by pumping to a pressure of 10^{-6} mm while the bulb containing the carbon dioxide was immersed in liquid oxygen. The oxygen contained about 500 ppm of nitrogen as the main impurity.

Results

Curves for the oxidation of zirconium in carbon dioxide at 550, 650 and 750 C are summarized in Figure 1, and shown in greater detail in Figures 2 and 3, as the weight gain in $\mu g/cm^2$ against the time in hours. At each temperature, the rate of oxidation first decreases and then increases with time. The weight gain at which this acceleration (i.e., breakaway) occurs *increases* with increasing temperature, the values being approximately 30 $\mu g/cm^2$ at 550, 60 $\mu g/cm^2$ at 650 and 180 $\mu g/cm^2$ at 750 C. Reference to Figure 2 shows that the two runs at 550 C are in agreement up to the time (about 16 hours) when breakaway occurs. After breakaway the rates of oxidation differ by as much as a factor of two (Figure 1). Moreover, the rate increases up to a maximum and thereafter decreases.

Corresponding curves for the oxidation of zirconium in oxygen at the same three temperatures are shown in Figure 4, and comparison of the data with those in Figures 1-3 shows that the weight gain after a given time in oxygen is considerably greater than in carbon dioxide at the same temperature (after 1 hr at 650 C, 588 $\mu g/cm^2$ compared with 37 $\mu g/cm^2$). This is contrary to the findings of O'Driscoll, Tyzack and Raine[8] who found the converse to be true.

A second and more important difference is that curves in oxygen do not show breakaway within the period of the experiments. Instead the rate of oxidation continuously decreases with time.

Previous work[2,4] has indicated that the oxidation of zirconium in oxygen can be described (up to the time at which breakaway occurs) by the approximate cubic rate law

$$w^n = kt \quad n \sim 3 \qquad (1)$$

where w is the weight gain at time t, and k is a constant. The present data were, apart from the first 5 min, found to conform well to this relation at 650 C in oxygen, and also in carbon dioxide, both at 650 and 750 C up to the time at which breakaway occurred (Figure 5). The values of k and of n are given in Table 2. At 550 C however, the conformity in oxygen was limited to the first 5 hours. In carbon dioxide the conformity could not be tested because breakaway intervened too early in the run.

The weight (w_c) of carbon deposited on the sample at the end of each run was estimated by direct counting using a Geiger-Muller tube with an end window, 2.5 cm in diameter. Counts were taken with the window placed over successive areas of the specimen. The reproducibility with a given sample was about 1 percent. The value of w_c was calculated from the measured count rate using a proportionality constant which had been obtained in some earlier work[12] dealing with the oxidation of beryllium in carbon dioxide. In the case of zirconium oxidation, it is believed that some of the carbon deposited on the oxide surface diffuses inwardly. In this case the values of w_c (Table 3) may be too low because of self-absorption (absorption half-thickness is about 8 μ) effects in the oxide. Subtraction of w_c from the total weight gain gives w_o, the weight of combined oxygen. As can be seen from Table 3, the ratio

TABLE 1—Analysis of Zirconium[1]

Element	ppm	Element	ppm
Hf	85	Ni	<20
Al	30	Pb	<20
B	0.3	Si	45
Co	<20	V	<20
Cr	130	Cl	300
Fe	930	O	<1400
Mg	550	N	80
Mn	<20	Ti	70
C	200	H	10

[1] The values for N, Ti and H refer to the sheet and the remainder to the sponge.

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* Obtained from Imperial Chemical Industries, England.

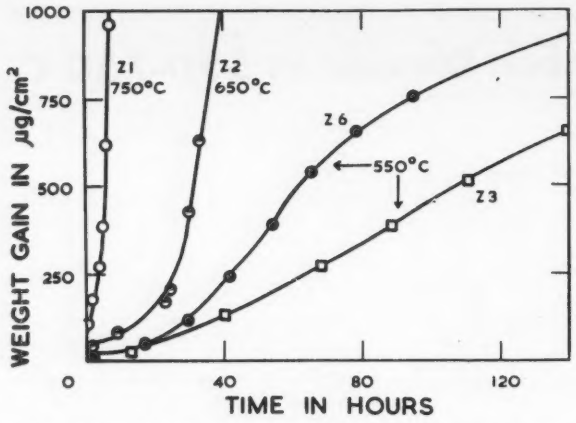


Figure 1—Oxidation of zirconium at 550, 650 and 750 C in carbon dioxide at 10 cm pressure.

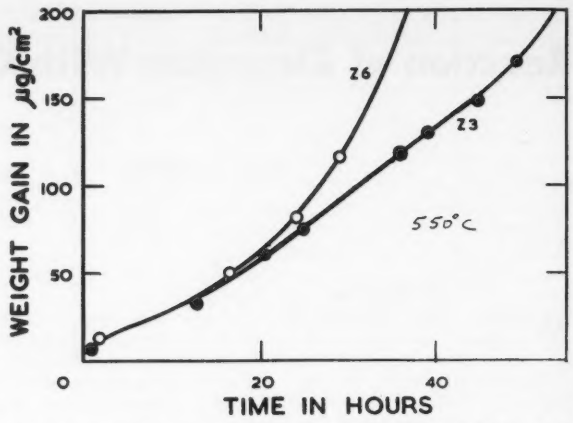


Figure 2—Oxidation of zirconium at 550 C in carbon dioxide at 10 cm pressure showing the first 50 hours of each run.

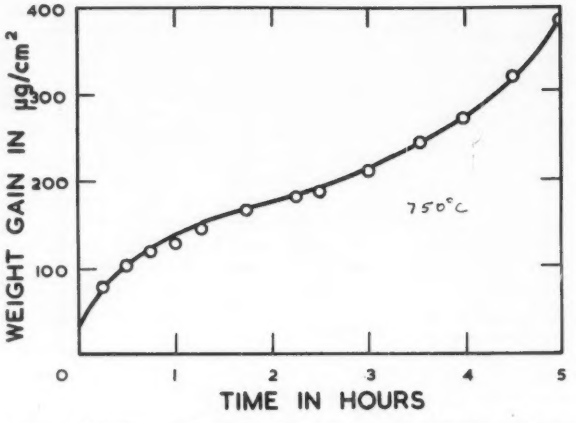


Figure 3—Oxidation of zirconium at 750 C in carbon dioxide at 10 cm pressure.

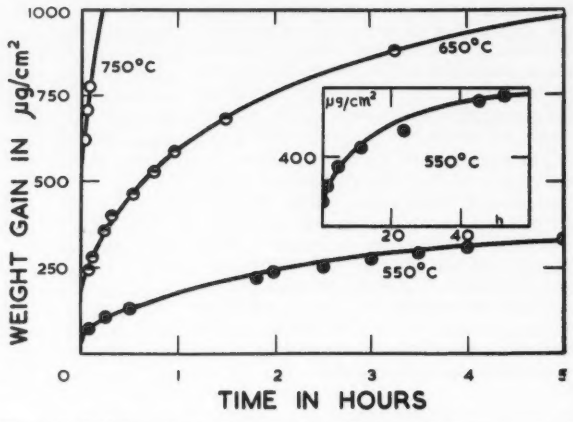


Figure 4—Oxidation of zirconium at 550, 650 and 750 C in oxygen at 10 cm pressure.

w_c/w_o is relatively small and shows no obvious trend with temperature.

The microbalance experiments give the total weight gain at successive points in time. It was hoped that with the use of a small volumetric apparatus developed for this purpose[13] corresponding data for the weight of carbon deposited could be obtained. The results of several runs were anomalous however, probably because of the relatively high rate of oxidation. As a result, the concentration of carbon monoxide (with which zirconium also reacts) soon built up in the apparatus. However, in one run a sample was oxidized in carbon dioxide at 650 C for 15 minutes on the microbalance and the weight of deposited carbon then determined after removal of the specimen from the balance. The value was considerably less than that obtained with the more heavily oxidized specimen (0.6 μg/cm² compared with 13.3 μg/cm², Table 3). This shows that carbon deposition occurs throughout the run and not merely at the beginning as is the case with beryllium.[11]

All the samples oxidized in oxygen and those oxidized at 550 C in carbon dioxide were bluish-black in appearance. Those oxidized at 650 and 750 C in carbon dioxide had a dull metallic appearance with some bluish-black patches. Autoradiographs[12] of the samples which had been oxidized in carbon dioxide showed that the deposition of carbon was fairly uniform at 550 C and somewhat less so at 750 C (Figure 6).

Discussion

From the value of the ratio w_c/w_o (Table 3) it is readily concluded that when zirconium is heated in carbon dioxide, approximately 98 percent of the measured weight gain results from the reaction

$$Zr + 2CO_2 = ZrO_2 + 2CO \qquad (2)$$

and the remaining 2 percent from one or both of the carbon deposition reactions

TABLE 2—Oxidation Rates of Zirconium in Oxygen and Carbon Dioxide*

Temperature (°C)	Gas	$(\mu g/cm^2)^n/hr$	n
650	Oxygen	6.40×10^7	2.82
	Carbon dioxide	2.51×10^5	3.46
750	Carbon dioxide	4.57×10^5	2.64

* Values of k and n in the equation $w^n = kt$.

TABLE 3—Weights of Deposited Carbon and Combined Oxygen Found in Oxidation of Zirconium

Run	Temperature (°C)	Time (h)	w_c (μg/cm²)	w_o (μg/cm²)	w_c/w_o (Percent)
Z6	550	90.5	19.5	732.9	2.7
Z3		157.0	11.6	712.6	1.6
Z2	650	33.0	13.3	616.2	2.2
Z7		0.25	0.6	15.1	4.0
Z1	750	7.75	14.2	1084.6	1.3

Note: w_c and w_o are the weights of deposited carbon and of combined oxygen respectively.

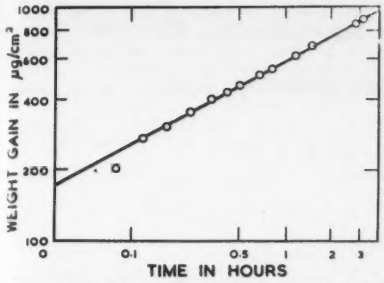


Figure 5—Oxidation of zirconium at 650 C in oxygen at 10 cm pressure. The logarithm of the weight gain is plotted against the logarithm of the time.

$$Zr + CO_2 = ZrO_2 + C \quad (3)$$

$$2Zr + CO_2 = ZrO_2 + ZrC \quad (4)$$

There is no significant trend with temperature in the values of w_c/w_o and therefore of the relative extents of reactions (2) and (3) [or (4)].

The balance of evidence[1,2,14-18] suggests that the oxide layer formed during the thermal oxidation of zirconium grows by the inward diffusion of O^{--} ions rather than by the outward diffusion of Zr^{4+} ion. Arguments based upon the relative sizes of the O^{--} ion and the Zr^{4+} ion[4] and experimental investigation[15] of the thermoelectric property of the oxide film suggest that the O^{--} ions diffuse between vacant lattice sites.

The physical significance of the cubic rate law which describes the present results for the oxidation of zirconium in oxygen, and in carbon dioxide up to the weight gain at which breakaway occurs, is not well understood. The several attempts[19,20] which have been made to provide a theoretical basis for this law have been based upon electrostatic field effects in p-type oxide layers.** These laws are applicable only to relatively thin films (several hundred Å thick) as compared with the thick oxide layers encountered in the present work.

Kofstad and Hauffe[21] have attempted to explain the cubic oxidation of titanium[22] (which like zirconium is capable of dissolving oxygen). This was done in terms of a model in which the rate of passage of oxygen from the oxide layer into the underlying metal is postulated to be equal to the rate at which it enters the oxide layer from the gas phase. The oxide layer thus remains of constant thickness, and the rate of weight gain is determined essentially by the rate of passage of oxygen through the layer of oxide. As far as the authors are aware the model has not been tested directly.

That the rate of oxidation at a given temperature is smaller in carbon dioxide than in oxygen suggests that the concentration of vacant anion sites at the gas/oxide interface influences the kinetics. An approximate calculation, assuming the absorption equilibria

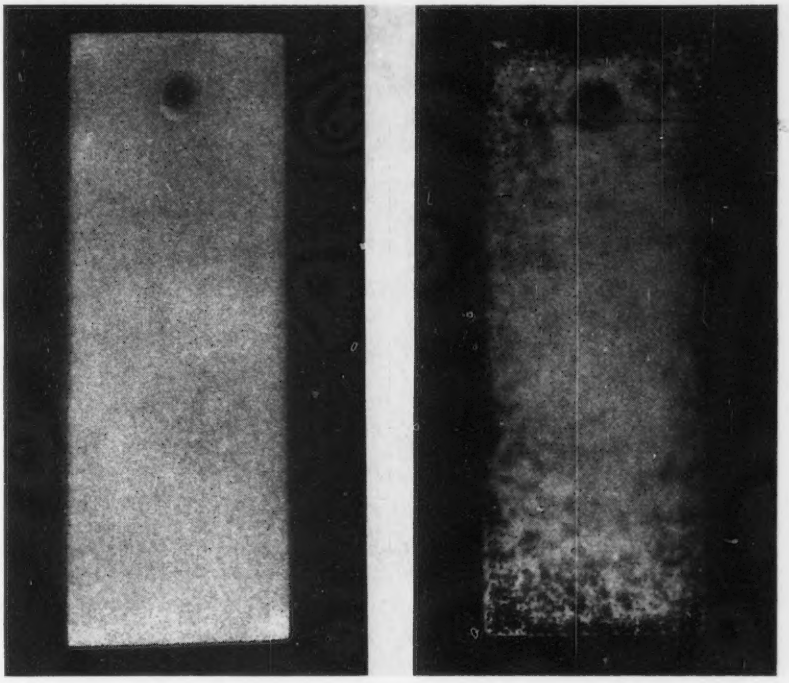
$$CO_2 + (O^{--}/\square) + 2(e/\square) \rightleftharpoons CO \quad (5)$$

and

$$\tfrac{1}{2}O_2 + (O^{--}/\square) + 2(e/\square) \rightleftharpoons \text{nil} \quad (6)$$

indicates that the concentration of vacant anion sites at the gas/oxide interface during oxidation in carbon dioxide exceeds that during oxidation in oxygen by a factor of 10^3. Because a high concentration of vacant oxygen sites is equivalent to a low concentration of occupied sites, the smaller rate of oxidation in carbon dioxide is in accord with the Kofstad-Hauffe[21] model.

**ZrO_2 is an n-type oxide.[14]



A—Run Z3, 550 C B—Run Z1, 750 C.

Figure 6—Autoradiographs of samples oxidized in carbon dioxide showing the distribution of carbon over the sample surface. Magnification 4x, reduced to 3x.

If it is accepted that the oxide layer on zirconium grows by the inward diffusion of oxygen ions, then it is clear that the carbon which is deposited at the gas/oxide interface according to the reaction:

$$CO_2 + 2(O^{--}/\square) + 4(e/\square) \rightleftharpoons C \quad (7)$$

will not become incorporated in the growing oxide layer as it would do if the metal were oxidizing by cationic diffusion. Guldner and Wooten[6] have shown that zirconium carbide is one of the products when zirconium powder is heated in carbon dioxide at 600-800 C. Because this carbon can be formed only where there is a source of Zr^{4+} ions, (i.e., at the metal/oxide interface), it would appear that there must be some inward diffusion of carbon atoms.

It seems probable therefore that the zirconium carbide is formed indirectly by the reaction

$$Zr + C = ZrC \quad (8)$$

rather than directly by the reaction

$$2Zr + CO_2 = ZrO_2 + ZrC \quad (9)$$

Perhaps the most striking feature of the present results is the contrast between the incidence of breakaway in oxygen and in carbon dioxide respectively. At 750 C for example, zirconium can be oxidized in oxygen to a weight gain of 800 $\mu g/cm^2$ without breakaway occurring, whereas in carbon dioxide breakaway takes place at a weight gain of only 180 $\mu g/cm.^2$ Even at 800 C, Porte and coworkers[4] did not observe breakaway in the 93-hr period of their tests in oxygen. However, Kendall, Wheeler and Bush[2] did find that breakaway occurred in air. The breakaway was delayed until the weight gain was 1000 $\mu g/cm^2$ at 500 C or 3000 $\mu g/cm^2$ at 700 C. The more ready occurrence of breakaway in carbon dioxide can very plausibly be attributed to the deposition of carbon and its diffusion into the oxide layer. Its presence there, whether in the elementary form or as zirconium carbide, would be expected to strain the lattice and thus promote film breakdown.

Finally, the fact that the weight gain at which breakaway occurs in carbon *dioxide* increases with increasing temperature may be interpreted in a manner similar to that suggested[23] for the oxidation of zirconium-tin alloys: the weight gain at breakaway is not simply a measure of the film thickness X but also includes the weight of oxygen which has diffused into the metal. Because of the positive temperature coefficient of this latter process, the value of X could in principle decrease with increasing temperature.

Conclusions

When zirconium is heated in carbon dioxide between 550 and 750 C, 98 percent of the total weight gain is due to the reaction

$$Zr + 2CO_2 = ZrO_2 + 2CO$$

and the remaining 2 percent to the carbon deposition reaction

$$Zr + CO_2 = ZrO_2 + C$$

The rate of oxidation first decreases and then increases with time indicating breakaway. In oxygen, on the other hand, the rate of oxidation continuously decreases with time and breakaway does not occur within the period of the experiments (maximum weight gain was 900 $\mu g/cm^2$). The breakaway in carbon dioxide is attributed to the deposition of carbon and its possible diffusion into the oxide layer. The weight gain at which breakaway occurs increases with increasing temperature of oxidation.

Acknowledgments

The authors are grateful to the United Kingdom Atomic Energy Authority for financial support.

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Any discussion of this article not published above will appear in June, 1962 issue

A Study of Scaling and Corrosion In Condenser Tubes Exposed to River Water*

By R. A. McALLISTER, DONALD H. EASTHAM, NEIL A. DOUGHARTY and MITCHELL HOLLIER

Introduction

Scope of the Problem

CONDENSER TUBE failure has increased sharply in the past several years at the Gulf States Utilities Company's Neches Power Station. This accelerated rate of failure was thought to result from an increase in the Neches River-water contamination accompanying local industrial expansion. The contamination is aggravated by the fact that the river-water flow rate has decreased by more than one-half in the past ten years.

The area power station is located on the Neches River approximately 15 miles upstream from Sabine Lake, which opens directly into the Gulf of Mexico. As a result of the low river-water flow, contamination of the river water by sea water is considerable.

The problem of studying corrosion and fouling of condenser tubes using Neches River water is complicated by the continually changing environment (i.e., fluctuations in the river water pH, temperature, hardness, chloride-ion concentration, dissolved oxygen, oxidizable organic material, etc.). Chlorine is added twice daily to the river-water before it enters the condensers, to inhibit biological growth. Waste caustic from demineralizers is added periodically to help inhibit corrosion.

Method of Study

In studying the over-all problem of condenser-tube failure, two phases of work were undertaken. The first was to try to identify organic contaminants in the river water that might be contributing to the increased corrosion. The second was to study the problem of corrosion in actual condenser tubes on the plant location, under conditions simulating condenser operation or differing from that only by certain controllable conditions. Only the latter course of study is discussed here.

In order to simulate Neches Power Station condenser operation, the experimental test apparatus was installed at the power station and constructed so that the same water which flowed through the actual condensers was used. Several separate tubes were installed and operated simultaneously. Certain tubes, referred to as control tubes, were operated at temperatures and river-water conditions identical to those of the plant condensers. For the other tubes in the test apparatus, certain variables could be controlled individually.

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Abstract

The series of tests reported give quantitative data on the rates of fouling and corrosion in 17 to 20 ft condenser tubes under simulated operating conditions using Neches River water as the cooling medium. Alloys tested were: 90-10 Cupro-nickel, aluminum-brass, Alclad aluminum, admiralty, three stainless steels, and alloy 77. Heat-transfer coefficients and Fanning friction factors were measured three times daily for 3 to 4 months. Several mechanisms of the fouling rate function were employed to compare the results. It appears that the heat-transfer coefficient is essentially linear with time and that the rate of fouling, initially at least, is not a function of velocity, but is a function of chloride-ion concentration (or hardness). The effects of weekly caustic addition, continuous cathodic protection, and river-water velocity were examined. Other river-water variables measured included pH, chloride-ion content, and hardness. The effects of these variables are discussed qualitatively.

3.3.2, 4.6.7, 7.4.2

Flow rate was considered to be an important variable in the study of fouling and corrosion. Thus tests were conducted using flow rates both greater and less than, as well as the same as, those in the plant condensers. Changes in velocity cause change not only in the quantity of corrosion and fouling agents supplied, but also the level of turbulence. The degree of turbulence is characterized by the Reynolds Number (N_{Re}).

Tubes made of various alloys were tested. A list of these is given in Table 1. In addition, some of the tubes were coated. Direct comparison could be made among different alloys as to the rate of change of the heat-transfer coefficient as corrosion and fouling progressed. The effectiveness of the tube coatings could also be determined qualitatively on a direct tube-to-tube comparison.

On one run a tube was cathodically protected with 10 milliamperes per square foot of tube surface.

At approximately 8-hour intervals, three times a day, readings were made on the test apparatus from which the heat-transfer coefficient and Fanning friction factor could be calculated.

TABLE 1—Alloys Tested

90-10 Copper-Nickel (Tubes 1, 2, 11, and 12)
Aluminum-Brass (Tubes 3 and 4)
Alloy 77, 70-30 Cu-Zn with special inhibitor (Tube 5)
Admiralty Brass (Tubes 6, 7, and 8)
Alclad Aluminum (data not graphed)
No 7-MO Stainless Steel (Tube 9)
No 20-CB Stainless Steel (Tube 10)
304 Stainless Steel (Tube 13)

Interpretation of Data

In addition to direct tube-to-tube comparisons of the results, it was desirable to interpret the data in such a way as to understand more clearly how fouling and corrosion occur. Corrosion and fouling in the condenser tubes are certainly simultaneous occurrences; in all probability the corrosion accelerates the fouling and, conversely, the fouling increases the rate of corrosion.

The formation of scale on the tube walls was determined by two methods in this study: by measuring the resistance of the tubes to heat transfer, and by measuring the pressure drop for flow through the tubes.

As the thickness of the scale builds up, the resistance to heat transfer increases. The equation used here to characterize the heat transfer is:

$$q = U_o A_o \Delta t_m. \qquad (1)^*$$

In this equation the over-all resistance to heat transfer may be written as the sum of several resistances,

$$1/U_o = R_o + R_w + R_i + R_s, \quad (2)$$

where R_o and R_i are the resistances to heat transfer of the fluid films on the outside and inside of the tube, respectively; R_w is the tube-wall resistance, and R_s is the resistance of the scale on the tube. As the fouling continues, the scale resistance would increase with time, while the other resistances should remain essentially constant.

McCabe[1] presented one of the earliest attempts to relate the heat-transfer coefficient to time. McCabe's equation was developed to describe the fouling of tubes inside an evaporator. From the assumption that the scale thickness was proportional to the total heat transferred, McCabe derived an equation,

$$1/U_o = b + a\theta \qquad (3)$$

McAdams,[2] following Seider,[3] suggests the use of a fouling factor, which is chosen sufficiently large to take into account the effect of the scale resistance, presumably for the life of the equipment. The fouling factor is not, nor was it intended to be, a function of time. Equation 2 becomes

$$1/U_o = (1/U_o)_{initial} + 1/h_d. \quad (4)$$

Both McAdams and Sieder give values of h_d for various types of environment.

Kern[4] has recently approached the problem by assuming that the scale thickness increased to some finite value asymptotically and in an exponential fashion. The resulting equation is

$$1/U_o = (1/U_o)_{initial} + R^* (1 - e^{-a\theta}), \qquad (5)$$

The mechanism which Kern proposes is that the rate of deposition of the scale is reasonably constant. As the scale thickness builds up, however, it is scoured away by the shearing action of the flowing fluid. Eventually the rate of removal of the scale equals the rate of deposition, so that the net rate of scale formation is zero (i.e., the scale thickness has increased to its constant value). The asymptotic value of the scale thickness would be an inverse function of the level of turbulence in the flowing fluid, since turbulence is one of the major factors determining the rate of scale removal. One would expect, therefore, to have a smaller final scale thickness at high velocities than at lower velocities.

It has been apparent from the beginning of this study that a linear scale build-up with time was a fair representation of the facts for most cases. Thus, if the thermal conductivity of the scale remained constant as the thickness increased, Equation (2) would become

$$1/U_o = (1/U_o)_{initial} + m\theta \qquad (6)$$

so long as the scale thickness is small compared to the inside diameter of the tube. The initial resistance to heat transfer, $(1/U_o)_{initial}$, is a function of: (1) the material and manner of construction of the tube, (2) the river-water properties of viscosity, density, etc., and (3) the velocity of the river-water flow inside the tube. If Equation (6) is applicable, the coefficient m describes the rate of increase of the resistance to heat-transfer and is directly proportional to the rate of build-up of scale.

It was anticipated that no such simplified equation as presented here would describe entirely the rate of change of the heat-transfer coefficient. It was expected that such factors as river water pH, temperature, chloride-ion concentration, and dissolved oxygen would be among the variables which would affect the rate of fouling and corrosion and, thus, the rate of change of the heat-transfer coefficient.

To describe completely the rate of change of the heat-transfer coefficient would require an equation involving all the variables which affect the corrosion and fouling, and not simply time. One of the purposes of this study was to discover the principal variables and to determine how they affected the rate of corrosion and fouling.

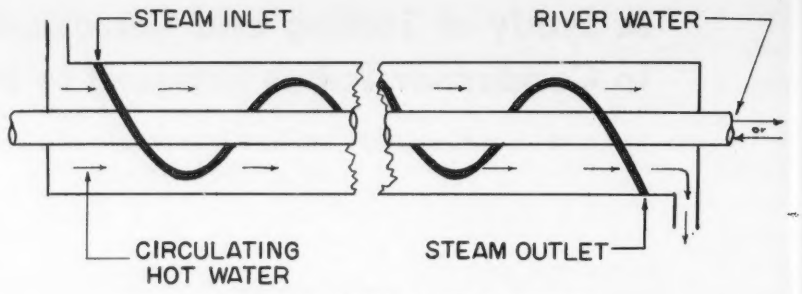

Figure 1—Diagram of a single test apparatus.

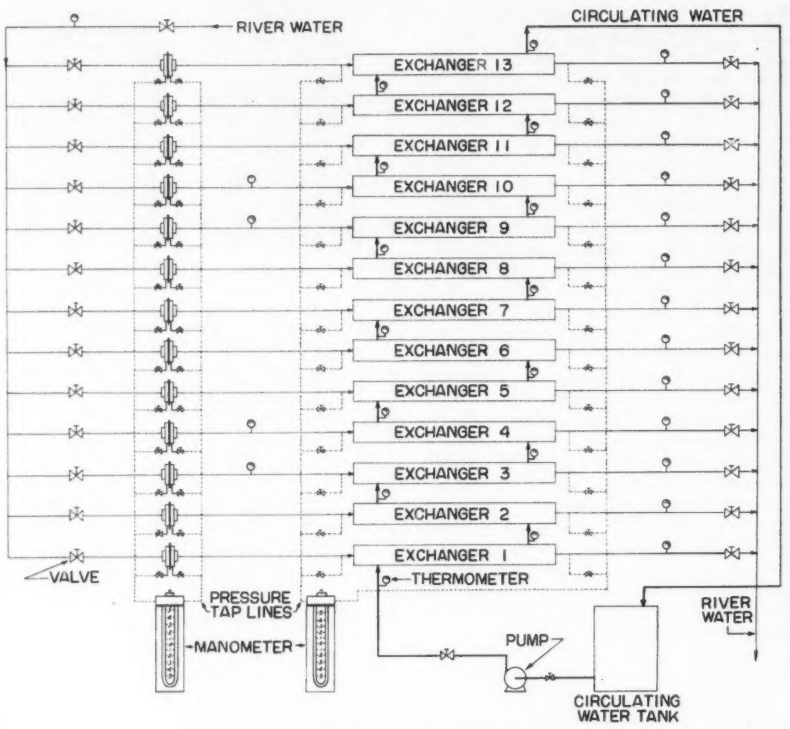

Figure 2—Over-all layout of the test apparatus.

*All symbols used are defined in the Nomenclature Section at end of article.

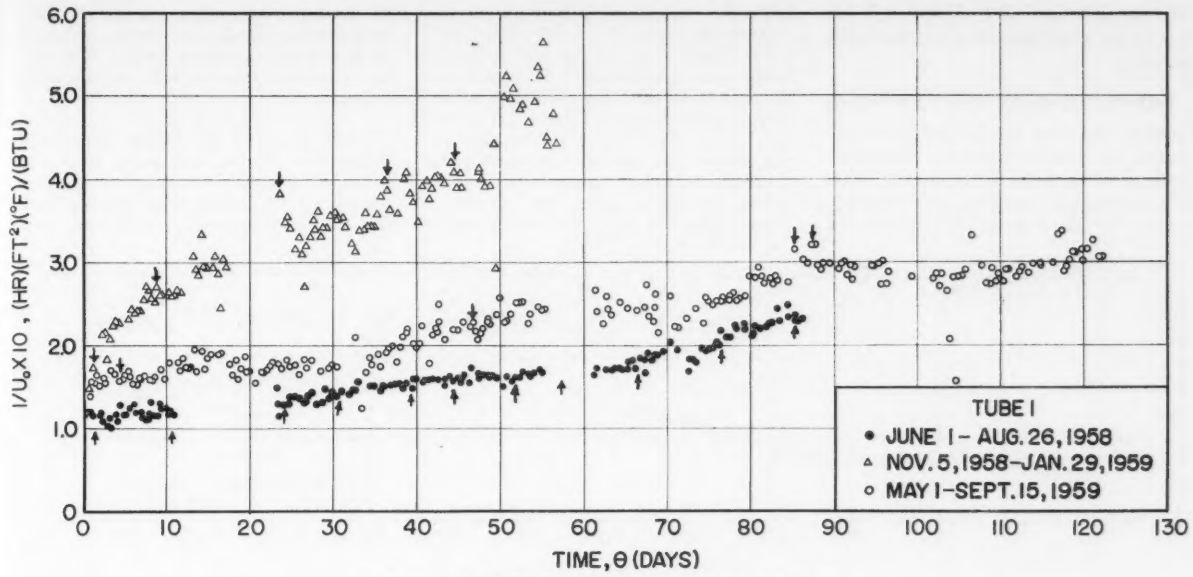

Figure 3—Heat-transfer resistance as a function of time.

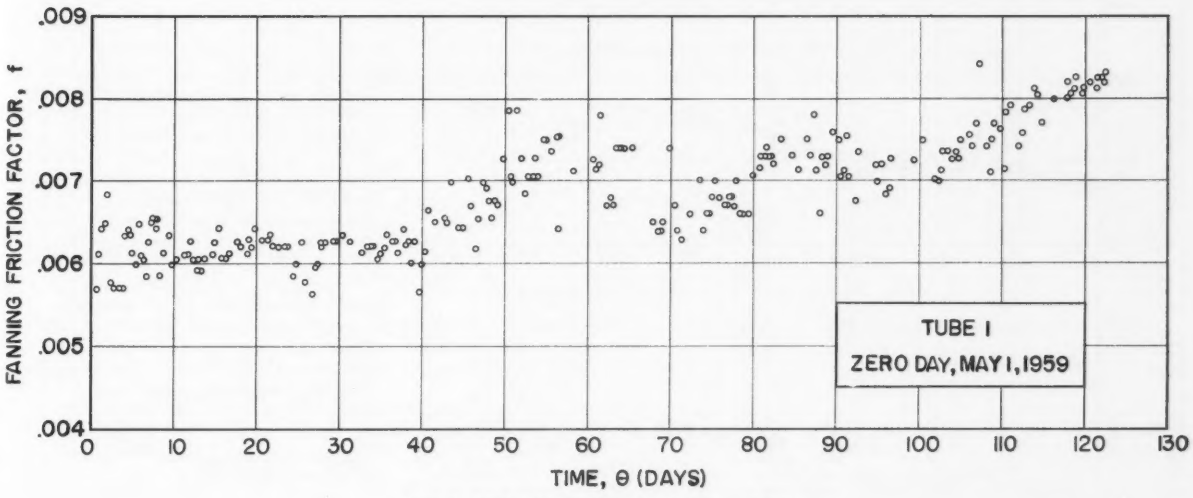

Figure 4—Fanning friction factor as a function of time.

Fouling was also characterized by measuring the loss in pressure of the river water in flowing through the tubes. Fanning friction factors were then obtained from the following relation:

$$f = (\Delta p)\, Dg_c/2LV^2\rho. \qquad (7)$$

All factors in equation 7 remain essentially constant except the pressure drop and the friction factor. The friction factor is a measure of the turbulent energy dissipation, and turbulence is affected by the roughness of the tube walls. The friction factors, therefore, are measures of the roughness and the amount of scale on the inside tube walls. In this study, the friction factors were found to be related to the heat-transfer resistances, as was expected.

Equipment and Procedure

A diagrammatic view of a single condenser tube exchanger is shown in Figure 1. The condenser tube was fitted with a removable concentric pipe jacket through which fresh hot water was circulated rapidly. In the annulus between the tube and the jacket, a copper tube carrying steam was coiled. The purpose of the steam tube was to maintain a constant heating temperature outside the condenser tube along its length, as there is in an operating condenser. Although the river water temperature rise was approximately 20 F, the circulating fresh water temperature was constant at 115 F, ±2 F.

Figure 1 shows the river water flow inside the tube to be either cocurrent or countercurrent to the fresh-water flow in the annulus, a situation which existed because of the over-all layout of the several tubes, which is shown in Figure 2. Figure 2 also indicates the positions of pressure taps, thermometers, orifices, etc.

The velocities in the test condenser tubes were maintained at 7.72 ft/sec, equal to that in the actual condensers, except for two tubes which were operated at 3.86 ft/sec, and 11.6 ft/sec, respectively. The velocities were measured by the use of orifices. Pressure drops over the lengths of the tubes were measured by a mercury manometer. All of the temperatures necessary to calculate the logarithmic mean temperature differences were measured using calibrated thermometers inserted directly into the flowing water streams. A record was kept of river water pH, hardness, and chloride-ion concentration. The pH was determined using a Beckman pH meter, hardness by the "Trilon B" method,[5] and chloride-ion concentration by the Mohr method.[6]

Results and Discussion

Qualitative Comparison of Alloys and Coatings Tested

In discussing the results in a qualitative manner, the alloys are discussed in three groups: (1) those containing cop-

per (the first four alloys listed in Table 1), (2) the aluminum, and (3) the stainless steels.

Performance of the Alloys Containing Copper. Insofar as this study is concerned, the copper-nickel, aluminum-brass, alloy 77, and admiralty brass tubes all performed in about the same manner. For the period May 1 to November 31, 1959, the resistance to heat transfer increased by about 3 x 10^{-3} (hr (ft^2) (°F)/(Btu). Tube 12, in which the velocity was approximately 1.5 times that of the rest of the tubes, had a considerably smaller increase, about 1.2 x 10^{-3} (hr) (ft^2) (°F)/(Btu). The Fanning friction factors for the tubes showed corresponding relative increases. Typical curves for these tubes are shown in Figures 3 and 4. The rates of change of the heat-transfer resistances and the Fanning friction factors are strong functions of river-water hardness and/or chloride-ion concentration, as will be discussed below.

Performance of Alclad Aluminum Tube. The Alclad aluminum tube was installed in the test apparatus in such a way as to be insulated electrically from the concentric heating jacket and from

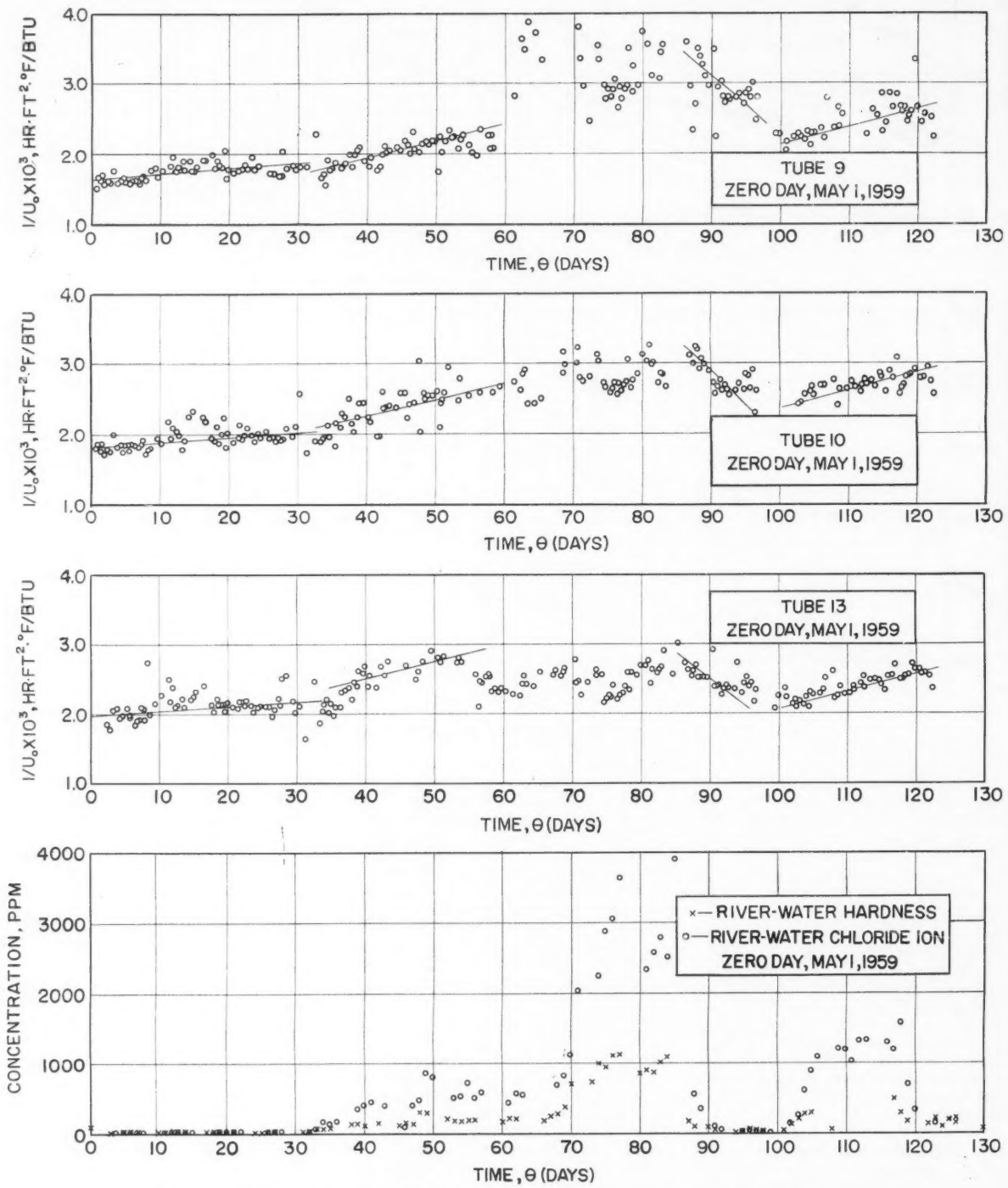

Figure 5—Heat transfer resistance and river-water chloride and hardness as functions of time for the stainless steels.

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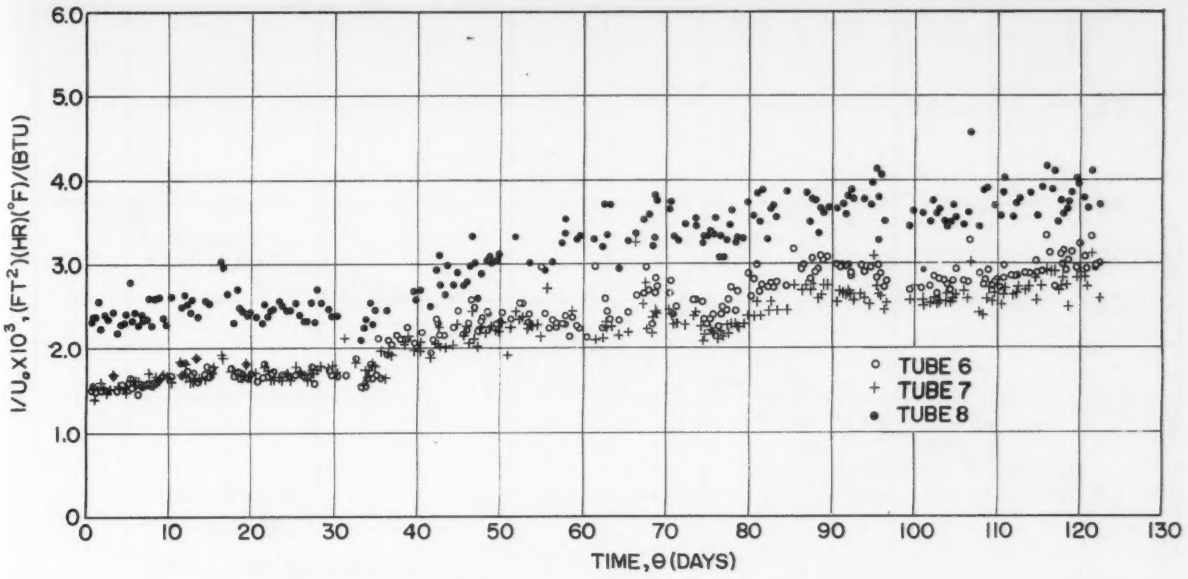

Figure 6—Heat-transfer resistance as a function of time, coated and uncoated admiralty brass.

the inlet and outlet headers. In this way strong galvanic corrosion due to contact of dissimilar metals was avoided. The aluminum tube deteriorated quite rapidly, however. In 22 days the heat-transfer resistance had doubled, and for the same time period the friction factor increased from 0.004 to 0.016, a factor of four. After about 60 days of operation, the tube was badly blistered and fouled.

Performance of the Stainless Steel Tubes. It will be noted from Figure 5 that the rates of increase of heat-transfer resistance were lower for stainless steel than for any other tubes operated at the same river water velocity. Though it will be shown below in the discussion of the effect of river water hardness and/or chloride-ion concentration that assuming a linear relation between $1/U_o$ and time does not give completely satisfactory results, the values obtained using this assumption do offer a reasonably satisfactory basis for tube-to-tube comparisons. Figure 5 shows that the heat-transfer resistance is a strong function of the chloride ion content (and/or hardness) of the river water.

The rates of change of heat-transfer resistance, m in Equation (6), as calculated by the least-squares method are given in Table 2. The values of m were 0.626×10^{-5} and 0.752×10^{-5} (ft²) (hr) (°F)/(Btu) (day) for the No. 7-MO (Tube 9) and the No. 20-CB (Tube 10) stainless steel tubes, respectively, while the corresponding values for the copper-alloy tubes averaged considerably above 1×10^{-5} (ft²) (hr) (°F)/(Btu) (day). The value of m for the 304 stainless steel tube (Tube 13) was even less, 0.260×10^{-5} (ft²) (hr) (°F)/(Btu) (day). Thus compared with the copper alloys, it is seen that the stainless steels performed somewhat better from the standpoint of heat transfer and fouling. This study did not attempt to characterize pitting of the tubes.

TABLE 2—Calculated Values of the Terms in Equation 6

Tube No.	(1/Uo) Initial	m	Correlation Coefficient	Std. Error of Estimate
1	0.001316	1.7146×10^{-5}	0.7293	0.0010106
2	0.001277	1.5872×10^{-5}	0.7160	0.0010573
3	0.001285	1.4672×10^{-5}	0.7850	0.0007131
4	0.001392	1.3756×10^{-5}	0.7188	0.0008173
5	0.001793	1.2033×10^{-5}	0.3288	0.0021490
6	0.001446	1.4833×10^{-5}	0.7416	0 0006121
7	0.001562	1.0385×10^{-5}	0.2391	0.0027597
8	0.002557	0.8507×10^{-5}	0.4246	0.0010121
9	0.001869	0.6259×10^{-5}	0.4380	0.0008044
10	0.001925	0.752×10^{-5}	0.8150	0.0003524
11	0.001930	1.8373×10^{-5}	0.3969	0.0026336
12	0.001592	0.4398×10^{-5}	0.4369	0.0003150
13	0.002133	0.2688×10^{-5}	0.5025	0.0003598

Effect of Tube Coatings. $1/U_o$, the resistance to heat-transfer, is shown in Figure 6 for three admiralty brass tubes as a function of time. Tube 6 was uncoated, Tube 7 was coated with a baked-on coating, approximately 3 mils thick, of a silicone dispersion in a phenolic, and Tube 8 was coated with a baked-on phenolic coating 5-7 mils thick. As seen in Figure 6, the silicone-coated tube behaved in a manner identical to that of the bare tube. The data for the phenolic-coated tube, though higher (as expected), closely paralleled the other data.

Several conclusions may be drawn from these observations. First, the resistance to heat transfer of the silicone coating was very small, if not negligible. The thicker phenolic coating resulted in an increased resistance and, hence, higher values of $1/U_o$. One point deserving of particular notice is that for three tubes the *rates* of fouling were identical. It is difficult to believe that any corrosion of note was occurring under the phenolic coating. The fouling which did occur, then, was a function not of the metal itself, but of the river-water conditions.

It is also unlikely that the inside surface temperatures of the tubes had much effect on the rates of fouling. This conclusion was drawn on the basis of the fact that the inside tube-wall temperature was always several degrees lower for the phenolic-coated tube than for the bare admiralty brass tube and yet the rates of fouling for these tubes were the same.

Qualitative Analysis of Heat-transfer Data

In Figure 7, the data for the heat-transfer resistance are given as a function of time for one copper-nickel control tube. The heavy line shown in the figure was calculated by the method of least squares.[7] For this particular line and set of data, the coefficient of correlation[7] is 0.720, and the standard error of estimate is 0.0010106. The coefficient of correlation and the standard error of estimate are almost meaningless when applied to the data obtained in this study, since these two quantities furnish a measure of the fit of data to a least square lines in the case where the data are randomly distributed about the line.

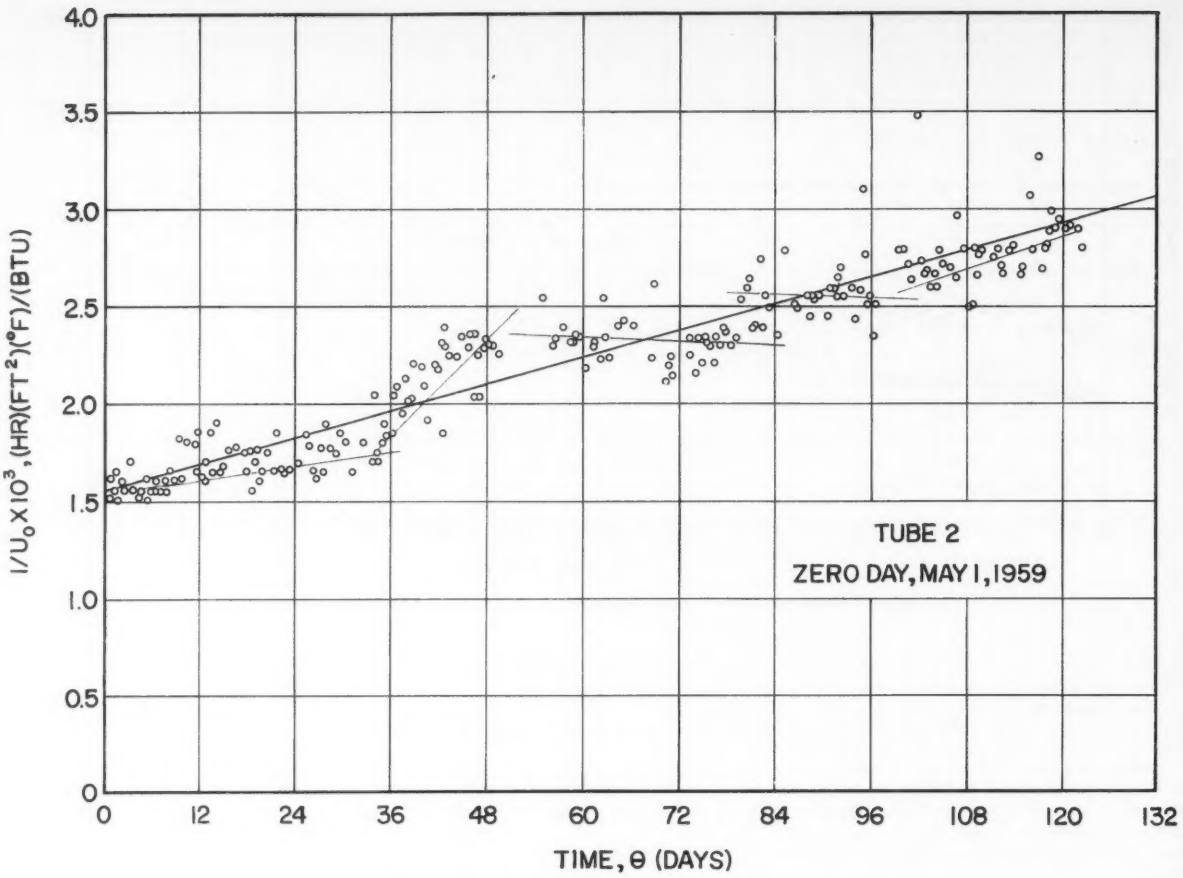

Figure 7—Heat-transfer resistance as a function of time.

That this is not the case will become apparent from the discussion below of the effect of river-water hardness and chloride-ion concentration.

Data for $1/U_o$ and friction factor versus time for the period May 1 to August 31, 1959, are tabulated for all the tubes in Reference 8. Table 2 gives a list of $(1/U_o)_{initial}$ and m for Equation (6), coefficient of correlation, and standard error of estimate for each of the 13 tubes tested during the period May 1 to August 31, 1959.

Since the $1/U_o$ data fit reasonably well a linear relationship with time over long periods in spite of the effect of chlorides, it does not seem justified to use an equation such as Equation (3), which relates $1/U_o^2$ to time. Under much higher rates of fouling such as one might expect to find in an evaporator, Equation (3) might offer a better fit for the data. For all the sets of data examined in this study a linear relation between $1/U_o$ and time is a satisfactory representation of the facts, except perhaps in the case of the one tube which was operated at high velocity. And, of course, the linear relationship is simpler to use. For the copper-nickel alloy tubes, the aluminum-brass tubes, and the uncoated admiralty brass tubes, operated at 7.72 ft/sec, the values of $1/U_o)_{initial}$ were from 0.00128 to 0.00179 (hr) (ft²) (°F)/(Btu). For the three stainless steel tubes, values were 0.00187 to 0.00213 (hr) (ft²) (°F)/(Btu.). The value of m in Equation (6) ranged from $0{,}268 \times 10^{-5}$ to 1.715×10^{-5} (hr) (ft²) (°F)/(Btu) (day) for the tubes operated at 7.72 ft/sec.

Effect of Velocity

The effect of three different river water velocities on the resistance to heat transfer is shown in Figure 8. All three tubes for which data are plotted are 90-10 copper-nickel alloy. In Tube 2 the river-water velocity is the same as that in the condensers at the Neches Power Station, 7.72 ft/sec. The velocity in Tube 11 is one-half that value, 3.86 ft/sec; and the velocity in Tube 12 is 11.6 ft/sec, about 1.5 times the normal operating velocity. The three lines shown in Figure 8 are those determined by the method of least squares. These lines give rather misleading pictures of the facts, particularly in regard to short periods of time within the total 130 day period.

Increasing the velocity of flow through a tube should improve the heat-transfer coefficient (i.e., $1/U_o$, the resistance to heat transfer, should be decreased). Conversely, decreasing the velocity should increase the heat-transfer resistance. Inspecting the data for the first 30 days, one finds from Figure 8 that the data fall approximately on three nearly parallel lines, with the line for Tube 12 below that for Tube 2 instead of these two lines crossing as the least-squares method shows.

The data for Tube 11, operated at low velocity, scatter considerably. It should be noted, however, that the trend of the data for this tube is about the same as that for the other tubes and that the rate of increase of the heat-transfer resistance is almost the same as that for Tube 2.

The data for Tube 12 give the only indication that the relation suggested by Kern's model, Equation (5), might be applicable to the results obtained in this study. Kern's model predicts that the scale builds up to a definite thickness asymptotically and exponentially with time. For the period from days 70 to 130 it is seen that the heat-transfer resistance is roughly constant. This seems to indicate that the rate of fouling is approaching the rate of removal of the scale by the action of the turbulent river water, as predicted by Kern.

The failure of Kern's equation to be applicable to the data generally is believed to result from one cause. As will be shown below, the rate of scale formation appears to be a strong function of river water chloride ion concentration and/or hardness. As a result, the assumption which Kern makes that the rate of deposition of the scale is essen-

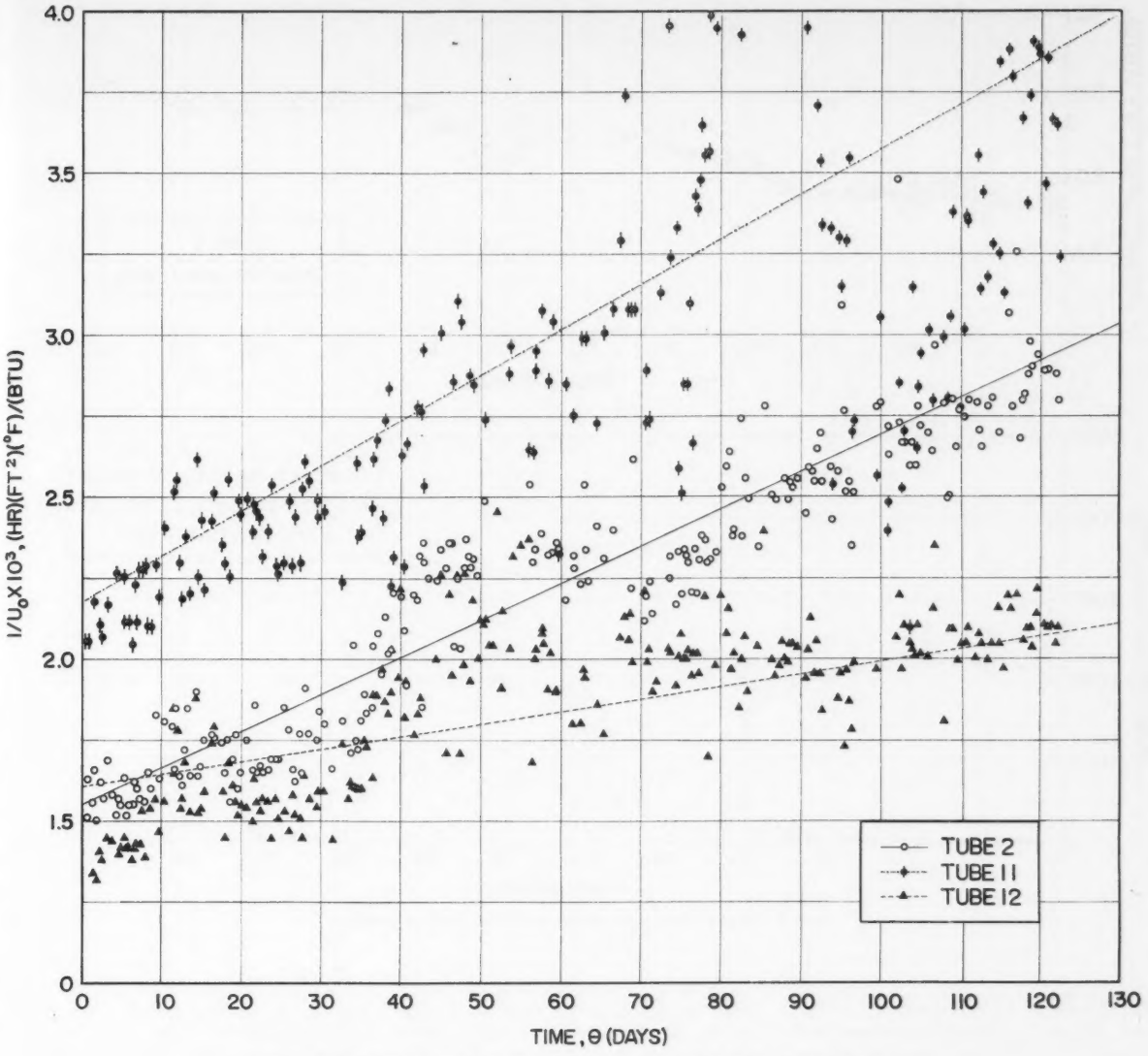

Figure 8—Heat-transfer resistance as a function of time at three river water velocities.

tially constant is not valid, and the value of R* would be continually changing with the river water conditions. It is also felt that the river water velocities used in the test apparatus were so low that the rate of scale removal was not sufficient to cause heat-transfer resistances to approach asymptotic values.

Effect of Chloride Ion on Fouling and Corrosion

Figure 9 shows the effect of chloride on a copper-nickel control tube while Figure 5 compares the heat-transfer resistance and chloride ion content with time for three stainless steel tubes. The data for Tube 1 are representative of the type of data obtained not only for all the alloys containing copper, but also for the stainless steels.

Particular attention should be given to the small lines drawn through the data of the upper graph in Figure 9. Notice that for the first 30 days the data fit well about the line drawn through the $1/U_o$ vs. Θ points. For this time period, notice also that the chloride ion concentration of the river water is very low. From day 30 to 60 the chloride ion concentration has risen to 500 or so parts per million. Notice that for the same time period, the rate of change of $1/U_o$ vs. Θ increases. That is, the line drawn through these points has a higher slope than for the first 30 days.

From day 30 to 85 or 90, the chloride ion concentration is rapidly changing first increasing to over 4000 ppm and then decreasing essentially to zero. During this period of time the heat-transfer resistance as well as the friction factor fluctuate widely. From day 90 to 100 during a period of almost no chloride ion in the river water, the heat-transfer resistance actually decreases, as seen by th line of negative slope shown in the upper graph of Figure 9. Then for days 100 to 120, again a period of changing but significant chloride ion content, 1000 parts per million of chloride, the heat-transfer resistance again rises with time.

Figure 3 shows heat-transfer resistance as a function of time for a copper-nickel control tube for three different runs. Examining Figures 9 and 10, showing river water hardness and chloride ion concentration during the three runs, one finds that the data for November 5, 1958, to January 29, 1959, on Figure 3 correspond to a period during which the chloride ion concentration ranged between 2000 and 5000 ppm throughout the run. During the first 60 days of the June 1 to August 26, 1958 run, the chloride ion concentration ranged below 1000 ppm; during the first 70 days of the May 1 to December 1, 1959 run, the chloride ion concentration ranged below 1000 ppm. Note that tubes of the same material were used on each of the runs, that the tubes were operated at the same river water velocities, and that the seasonal periods (and hence the river-water temperatures) overlap. Yet the rate of increase of heat-transfer coefficient is sev-

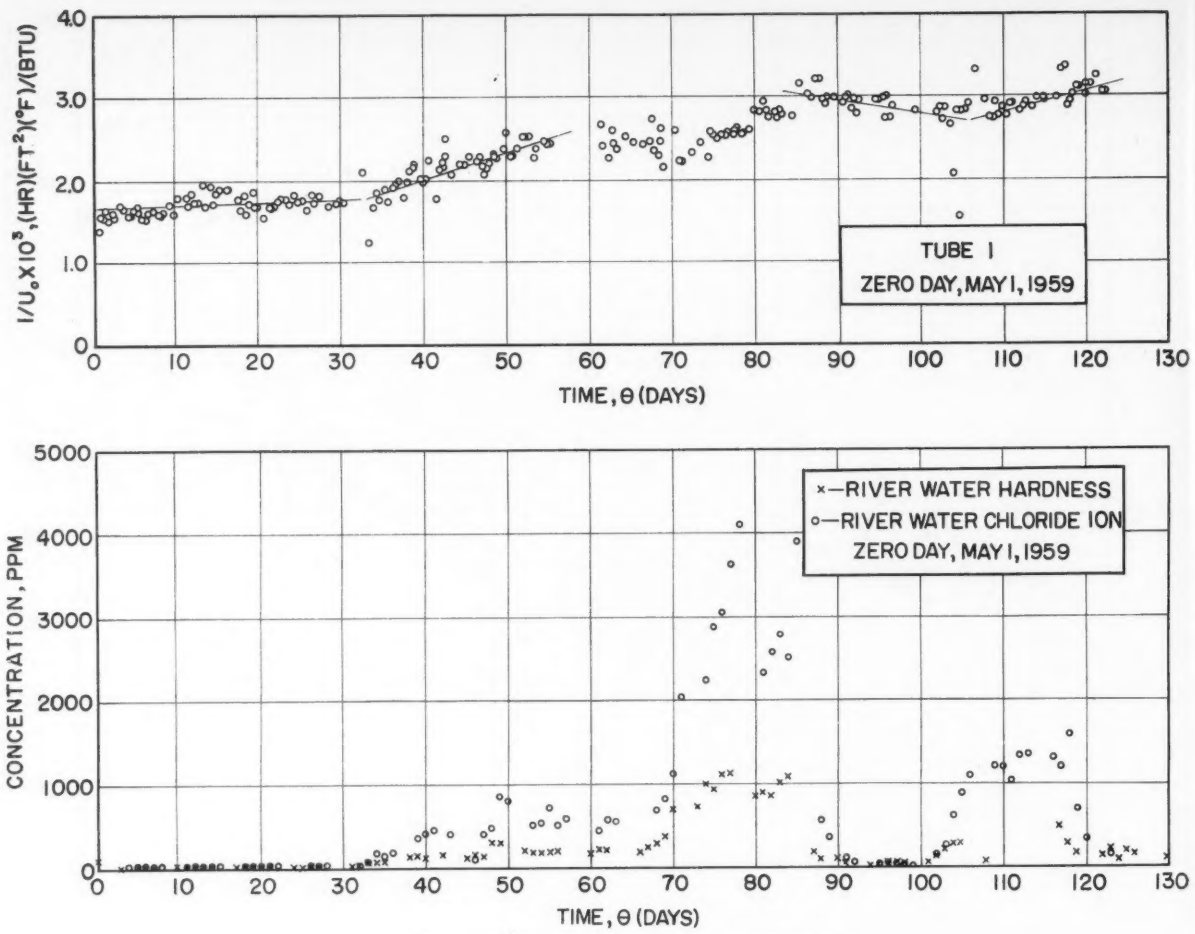

Figure 9—Effect of chloride ion content on the rate of change of fouling.

eral times greater (about 5 times) for the November 5, 1958-January 29, 1959 run, (the period of high river water hardness and chloride-ion concentration), than for either of the other two runs. Note also that the data for the two runs made during periods of relatively low chloride-ion concentration and hardness lie roughly on two nearly parallel lines.

For the stainless steels Figure 5 shows the same trends. As the chloride concentration increases, the rate of fouling increases (compare the data for the three tubes for the period 30 to 60 days, and 100 to 120 days). During the period of high chloride, 3000 to 4000 ppm, the data for stainless steel are somewhat erratic, except for Tube 13 which is the 304 stainless steel. In the period from 85 to 100 days where the chloride ion concentration in decreasing to zero, the resistance to heat transfer diminishes.

All these observations, for both the copper alloys and the stainless steels, lead one to conclude that river water chloride ion concentration and/or hardness are major determining factors in the rate of fouling. The data at present afford no possibility for determining the relative effects of these two factors, since the hardness parallels exactly the chloride-ion concentration.

One further observation needs to be made in regard to the rates of fouling. For all of the alloys containing copper and operated at 7.72 ft./sec., the rates of fouling were roughly the same (see least-squares values for m of Equation (6), in Table 2). For the No. 7-MO and No. 20-CB stainless steel tubes, the rates of fouling were somewhat lower, slightly above half the average of that for the copper alloys. And for the 304 stainless steel, the rate of fouling was only about one-fifth that for the copper alloys. Initially, for the period from 0 to 30 days, when the hardness and chloride-ion concentration were low, the rates of fouling were about the same on both the copper alloys and the stainless steels. Only after the hardness and chloride-ion concentration rose was there any obvious differences in the behaviors of the two classes of metals and among the stainless steels themselves. From the standpoint of fouling, the stainless steels definitely performed better than the copper alloys and the 304 stainless steel seemed considerably superior to all the rest of the tubes.

Effect of Miscellaneous Variables

The change in the river water pH was very small, varying usually only between 6.8 and 7.1 except during periods of caustic addition. For such a small variation in the pH of the river water it was not possible to detect any resulting change in the operating characteristics of the tubes.

When caustic was added, the pH increased to as much as 9 to 10 for perhaps half an hour. There was no immediately detectable change in the heat-transfer resistance or in the friction-factor curves following periods of caustic addition. This may be seen in Figure 3, in which the small vertical arrows indicate the days on which caustic was added. From this it can be concluded that the caustic addition neither dissolves the scale nor causes more scale to deposit than normal. If either of these occurred, there would be a noticeable change in the heat-transfer resistance and friction-factor curves.

It is quite possible that the heat-transfer and friction-factor data are simply inadequate to show the effect of caustic addition. Plant operating data show that the caustic addition increases the life of the tubes. The mechanism by which this is accomplished is not known. It has been suggested that perhaps there is a decrease in the severity of pitting under conditions of caustic addition. Plans are laid to investigate this possibility and

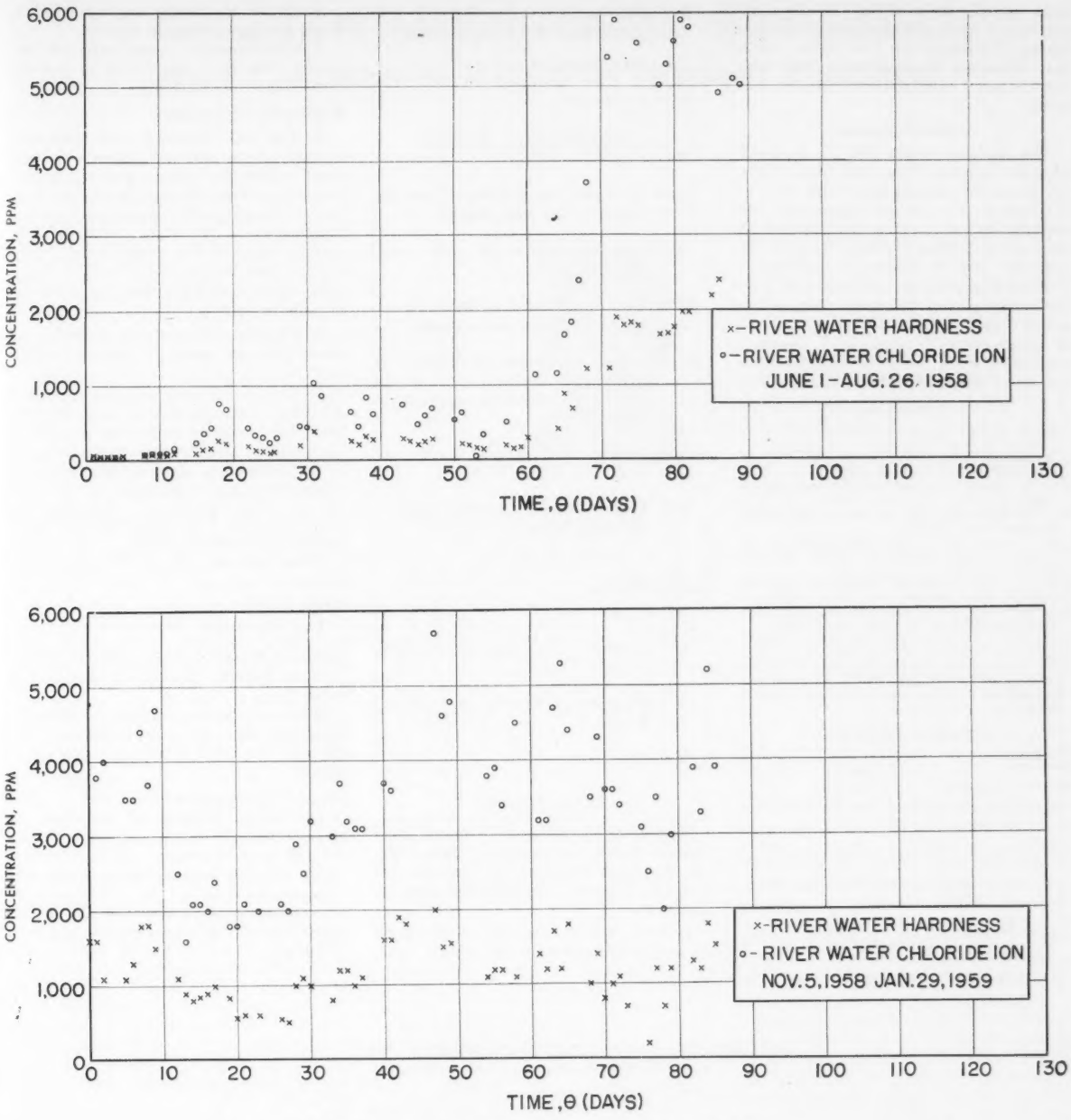

Figure 10—River-water chloride and hardness as a function of time.

characterize pitting, both as to the number of pits per unit area and as to the size distribution and depth of the pits.

The rate of fouling was not noticeably affected by the cathodic protection which was attempted. Here, also, it will probably be necessary to evaluate the pitting.

Conclusions

1. Under river water conditions where the chloride ion concentration is reasonably constant, the heat-transfer resistance, $1/U_o$, is approximately linear with time.

2. Increase in the river water chloride ion concentration (accompanied by a simultaneous increase in hardness) results in an increased rate of fouling as measured by the slope of the $1/U_o$-versus-time curves. A sustained decrease (for several days) in the chloride ion concentration following period of high concentration results in a decrease in the scale thickness, again as measured by the decreased resistance to heat transfer, $1/U_o$.

3. Cathodic protection of 10 milliamperes per square foot had no detectable effect on the rate of fouling for copper-nickel and aluminum-brass condenser tubes using the Neches River water that was available during the test period (4000-6000 ppm chloride ion).

4. The rate of fouling at a velocity of 7.72 ft/sec was approximately the same for copper-nickel, aluminum-brass, Alloy 77, and admiralty brass. For the equation,

$$1/U_o = (1/U_o)_{\text{initial}} + m\Theta,$$

where $1/U_o$ has the units of (hr) (ft^2) (°F)/(Btu) and Θ has units in days, m for these tubes was about 0.000014 ± 0.000002.

5. Weekly additions of caustic had no effect on the rate of fouling. It is not possible with the heat-transfer data and friction-factor data alone to determine fully the effect of caustic addition.

6. Change in the river water velocity over the range from 3.86 ft/sec to 11.6 ft/sec does not appreciably alter the initial net rate of fouling under the condi-

tions tested. After about 20 days, the net rate of fouling decreased for the tube having a velocity of 11.6 ft/sec, while that remained approximately the same for the tubes operated at 7.72 and 3.86 ft/sec.

Acknowledgments

The authors would like to acknowledge the support of the Gulf States Utilities Company, and in particular of Wm. B. Gurney. The authors appreciate the opportunity of having participated in such an extensive and fundamental study, and they wish to thank the Gulf States Utilities Company for permission to publish the results of the study. Finally the authors want to thank the many students of Lamar State College of Technology who spent many hours carefully gathering, calculating, and presenting the data.

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NOMENCLATURE

Symbols

a, b = constants

A_o = outside area of the heat-transfer surface, ft^2

D = inside diameter of the tube, ft

e = the base of natural logarithms, 2.71828 . . .

g_c = Newton's Law conversion factor, $lb_m(ft)/(lb_f), (sec^2)$

h_d = fouling factor for scale, a constant, Btu/(hr) (ft^2) (°F)

L = tube length, ft

m = a constant

N_{Re} = Reynolds Number = $\frac{DV\rho}{\mu}$

Δp = decreased in pressure over the length of the tube, lb_f/ft^2

q = heat transferred per unit time, Btu/hr

R^* = asymptotic value of the scale resistance to heat transmission

Δt_m = log mean temperature difference between the heating water on the outside and the river water on the inside, °F.

U_o = over-all heat-transfer, coefficient, Btu/(hr) (ft^2) (°F)

V = average velocity of the fluid in the tube, ft/sec

Greek Letters:

α = a constant

μ = absolute viscosity of the fluid, $lb_m/(ft)$ (sec)

ρ = density of the fluid flowing, lb_m/ft^3

Θ = time, days

DISCUSSION

Comments by Sidney E. Trouard, New Orleans Public Service, Inc., New Orleans, La., and by Lloyd O. Timblin, Jr., U.S.B.R. Denver Federal Center, Denver, Colorado.

1. What was the nature of the scale formation described in the paper?

2. Could the water be described as being brakish or highly saline?

3. Corrosion-wise, what type of condenser tube have you found to perform best under the conditions described?

Replies by the authors:

1. The scale formation was primarily cuprous oxide, although there were also small amounts of sulfides and iron oxides. During periods of high hardness, there were undoubtedly some calcium and magnesium in the scale. Near the tube surface, there was a rather hard chalky scale, while on top of it was a black, slimy, gelatinous scale that was primarily organic in nature.

2. The chloride content ranged from essentially zero parts per million to 6000 ppm.

3. The results show only the combined effects of corrosion and fouling. On this basis either the stainless steel tubes, or the silicone-coated tubes might be said to be better. The data, however, do not reveal the effects of pitting.

Question by F. B. Hamel, Midland Building, Cleveland, Ohio:

What was the circulating water temperature?

Reply by the authors:

The temperature was 115 F.

Question by Howard J. Cohan, U.S.B.R., Denver Federal Center, Denver, Colorado:

What was the composition of dissolved solids in the water and why was the chloride ion chosen as the variable to follow?

Reply by the authors:

The actual analysis of the dissolved solids was not determined, other than chloride and "hardness" (see text). Actually chloride and hardness concentration paralleled each other. This variable was chosen since it was the one which seemed related to the change in the rate of fouling.

Any discussion of this article not published above will appear in June, 1962 issue

Corrosion Problems in the Use of Zirconium And Columbium Alloys in Nuclear Reactors*

By D. L. DOUGLASS

Abstract

Corrosion problems relating to the use of zirconium in reactors are discussed in terms of corrosion rate transition and hydrogen pickup. The phenomenological aspects and the resultant practical implications are related to limitations in reactor performance. The effect of irradiation is shown to be important and not too well-defined. The rate of hydrogen pickup and its interaction with radiation damage and structural characteristics of zirconium are discussed with respect to degradation of mechanical behavior. Justification of columbium can be achieved only for superheat steam or liquid metal-cooled reactors. The kinetics and phenomenology of high temperature steam corrosion are presented. The limiting factor appears to be oxygen contamination and subsequent embrittlement due to rapid diffusion of oxygen at superheat temperatures. The oxygen sensitivity of liquid metal corrosion appears to be limiting for space power nuclear reactors. 8.4.5, 6.3.20, 6.3.5

Introduction

THE LIMITING factor in the operation of nuclear reactors is not due to engineering design or construction but rather to the limitations in the materials employed. The materials themselves are adequate and should perform well in their initial condition at the startup of the reactor, but unfortunately these materials change during the course of operation from both the effect of radiation and corrosion by the coolants used. It is the changes which are induced by the corrosion process which are responsible for the deterioration of the initially well-behaved engineering materials. These changes ultimately result in limited lifetimes and performance.

The number of materials available to the reactor design engineer is very small, and for those in the commercial aspects of the business who are endeavoring to make nuclear power competitive with fossile-fired power plants the problem is severely compounded. Cost is an object, and one is therefore faced with decreasing the costs of reactor components as well as improving operating efficiencies and lifetimes. Certain possibilities are thus immediately obvious: (1) substantially increase the lifetime of fuel cladding, pressure tubes, channels, etc., (2) increase the operating temperature to give higher efficiency, (3) decrease the cost of the base materials and fabrication costs, or (4) select alternate materials.

One of the first considerations must of necessity be that of neutron capture cross section. For boiling water or pressurized water reactors the number of base metals on the basis of cross section which have sufficiently high strength (when alloyed) and a fair degree of corrosion resistance is limited to zirconium, columbium, and iron base alloys. If one limits the discussion to just corrosion behavior and the degradation of mechanical properties resulting from the corrosion process, he can see that a definite upper limit exists for the use of zirconium alloys in this type of reactor. Zircaloy-2 has proved very useful for so-called low temperature applications in which the ambient coolant temperature is about 550 F and the maximum cladding temperature is about 750 F. Higher steam temperatures prove catastrophic to Zircaloy-2, and a new alloy must thus be developed for higher operating temperatures.

Earlier work at Nuclear Metals showed some promising zirconium alloys at temperatures of 950 to 1050 F steam, but these results must be tempered with the realization that spherical symmetries were utilized and may have been responsible in preventing spalling of the protective oxide coating. Subsequent work performed at other sites has not resulted in zirconium base alloys capable of sustained performance at these temperatures. It can be concluded that zirconium alloys cannot be developed to withstand these temperatures for periods of several years unless a radical breakthrough is achieved, and this does not appear promising.

The remaining materials, columbium and iron, both have substantially higher cross section than zirconium, and their use would cause a large sacrifice in neutron economy. Justification would therefore have to be on the basis of much higher operating temperatures and longer lifetimes. Further, the cost of columbium presently is too high to permit its use. It is generally thought, however, throughout the refractory-metal industry that the cost will drop over the next few years to the extent that it will cost no more than zirconium. The factors which preclude the use of zirconium in the next generation of high temperature reactors and the factors which may be limiting in the unmitigated use of columbium are worthy of examination.

About the Author

D. L. DOUGLASS, is senior metallurgist on the Vallecitos Atomic Laboratory Staff, General Electric Company, Pleasanton, Calif. He was recently transferred from the Knolls Atomic Power Laboratory where he was Supervising Metallurgist, Alloy Studies. Dr. Douglass was formerly associated with both Battelle Institute and DuPont at the Savannah River Laboratory. He received BS and MS degrees from Pennsylvania State University and a PhD in Metallurgy from Ohio State. Dr. Douglass, also a member of the faculty at Ohio State, served as chairman of the 1960 AIME Columbium Symposium and was editor of the conference proceedings. He has published a number of papers on uranium alloys, zirconium alloys, and on columbium alloys. He is a member of AIME and ASM.

Zirconium

The corrosion problems in zirconium can be broken down into two general categories and will be treated individually although the two categories are not independent. In addition, it should be emphasized that what constitutes a problem for one set of conditions does not necessarily constitute a problem for another set of conditions.

Corrosion Transition

The initial corrosion process in most zirconium alloys follows a logarithmic, cubic, and/or parabolic rate law. After a finite period the corrosion process undergoes a change which is observable as a linear rate law. A plot of log weight gain versus log time exhibits a change in slope at the transition point which separates the two straight line portions. The time for transition is affected by many variables such as composition, temperature, environment, and microstructure. These factors are generally well known, therefore the examination of the phenomenological behavior and relation of this to the practical implications are necessary.

The initial step is essentially a diffusion-controlled process in which the increase in thickness of the oxide film with respect to time is inversely proportional to the thickness. The corrosion product, the oxide film, physically separates the two reactants, metal and water (or steam). Additional reaction is then dependent upon the migration of one or both of the reactants through the barrier. However, a process which obeys a linear rate law is one in which no barrier exists between the reactants. The amount of reaction is directly proportional to the time. In order for a protective barrier, such as the initial oxide films, to become non-protective and effectively eliminate the barrier between reactants, a degradation or breakdown in the film must occur. An interesting aspect of this problem is that in most cases no visual spalling or breakdown of the film is observed. Cox[1] has observed microcracks in Zircaloy-2 at grain boundaries by the use of electron microscopy, but oddly enough cracks were observed at times corre-

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sponding to pre-transition. One cannot say unequivocally that transition occurs when the oxide film cracks. Further, the dark film generally associated with protective conditions still exists at post transition.

The fact that black oxide still exists after transition implies that cracking of the oxide parallel to the surface does not occur. If parallel cracking took place, one would expect to see white patches of oxide over the crack due to the effective discontinuity which would then exist in the path of oxygen migration to the oxygen sink, the substrate metal. The only plausible mechanism which can account for all the observations, and as yet, this mechanism is unproved for zirconium, is the one proposed by Webb, Norton, and Wagner[2] on the parabolic to linear transition in tungsten and later generalized by Haycock.[3] A barrier scale forms at the outset and reaches a limiting, constant thickness because of an equality between the rate of formation and the rate of depletion by a secondary process on the exterior of the film. The secondary process is considered to be recrystallization of the oxide which results in the formation of an outer porous or "open" film. The cracks are perpendicular to the surface and generally offer paths of easy access for the reactants, in this case the inner oxide and the corrosion atmosphere. The mechanism applies for metals which form either a single oxide or multiple oxides.

One last observation should be mentioned which further complicates the picture. Thomas and Chirigos[4] investigated the oxidation of zirconium in both water and oxygen gas and observed that transition occurred in water but not in oxygen during the times employed. If transition were due to the strains corresponding to a given film thickness, then film breakdown should occur at the same oxide thickness regardless of whether the process occurred in water or oxygen. It appears that the hydrogen exerts a dominant but unknown role.

Another aspect of transition is the effect of hydrogen migration either in or out of the film.[5] Alloys which had been corroded for some time less than that to cause transition, removed from test, charged with hydrogen and retested, show immediate transition at times which ordinarily did not cause this behavior. Likewise, samples which were initially charged with hydrogen, corroded for times less than required to produce transition, removed from test, subjected to vacuum discharging of the hydrogen, and retested, showed premature transition. The migration of hydrogen damaged the film in some manner which permitted the parabolic corrosion reaction to change to the linear reaction.

TABLE 1—Time for Transition for Zircaloy-2

Temperature	Medium	Time for Transition, Days
550 F	Water	1800 ± 200
680	Water	110
750	Steam	40
850	Steam	10
900	Steam	2

Having looked briefly at the phenomenological aspects of transition, one may examine now the significance of transition from a practical point of view. Aside from the problems attributed to hydrogen pickup, rapid corrosion at high temperatures at post transition times results in a much larger loss of metal than for a similar exposure under the slower, diffusion-controlled, corrosion conditions. Structural or cladding components having thin sections will be either literally consumed by the corrosion process, or the reduction in cross-section will cause an increase in stress for a constant load and ultimate mechanical failure. The net result is a decreased component lifetime. Heavy sections are not seriously affected provided the reaction rate is low enough to prevent a high fraction of total metal from being consumed, but metal loss is very important in thin sections.

How does one combat this state of affairs? An obvious solution is to select operating temperatures which are low enough to insure that transition does not occur during the expected lifetime. The time for transition is an inverse exponential function of temperature, some representative values being shown in Table 1. If Zircaloy-2 were to be used for an expected lifetime of about 5 years under pretransition conditions, the temperature would be limited to about 550 F.

A second, less obvious solution, is to use a zirconium alloy having longer times to transition than Zircaloy-2. Presently, there are no commercial zirconium alloys available having both longer transition times and other properties superior to Zircaloy-2. Much effort is being expended on various alloy development programs, but as yet researchers are still faced with an empirical approach to solve the problem by techniques other than by numerous tests.

The simple fact that higher corrosion rates consume more metal is not, unfortunately, the only ramification of transition. Hydrogen pickup rates vary between pre- and post-transition times, and although more corrosion produces more by-product hydrogen which is available for pickup, the hydrogen problem is more complex than is apparent on the surface. Amplification of this statement will be made under hydriding problems.

Perhaps the most important consideration of all is the effect of irradiation. Differences have been noted between laboratory or loop corrosion tests and corrosion behavior under irradiation.[6] Exact measurements are not available, but it may be concluded that in irradiation service corrosion rates at a given temperature are higher than out-of-pile tests indicate. Cox[7] has shown that beta and gamma rays have little, if any, effect on corrosion, but fission fragment recoil is significant. Yee[8] has also established this fact under slightly different conditions, and Jenks[9] reports similar behavior in the presence of fissioning uranyl sulfate solutions.

The problems resulting from radiation can be narrowed down to two direct effects according to Biefer et al.[10] These are, (1) radiolytic decomposition of water, the presence of free radicals causing an acceleration in both rate and transition, and (2) radiation-induced damage to the film. Jenks[9] feels that the radiation damage is by far the dominant factor of the two. From an intuitive point of view it would appear that fission fragments would cause film damage, creating additional defects, etc. Noggle and Stiegler[11] have noted fission tracks in thin foils of UO_2 irradiated to about 10^{14} to 8×10^{15} nvt. Bierlein and Mastel[12] have observed fission damage tracks in thin films of ZrO_2 in which uranium was not present. An exposure of 2×10^{16} nvt was sufficient to cause large numbers of these defects. It is not unreasonable to expect extensive damage in ZrO_2 film during normal reactor exposure of 10^{21}-10^{22} nvt.

Two other indirect effects must also be considered. These are the higher corrosion rates which exist under heat transfer conditions, and the increase in temperature of the fuel element surface due to "crud" buildup.

Hydrogen Pickup

The entry of corrosion by-product hydrogen into the zirconium metal appears to be more of a limiting factor than does the corrosion itself. All of the aspects of this problem and the physical and chemical metallurgy associated with it have been covered in two excellent reviews. Shannon[13] summarized the available information up to April 1958, and Biefer et al[12] up-dated this area through September, 1959.

Much data have been reported in the literature on hydrogen pickup fractions for various alloys, the effect of temperature on hydrogen pickup, and the effect of various atmospheres on pickup. Rather than reiterate much of what has been said already, one may look first at the practical implications of hydrogen entry into zirconium during reactor operation and conclude this subject by examining the variation of pickup rate and its relation to transition.

The pickup fraction itself means very little because of variation in sample geometry. The actual concentration and its distribution are important in view of the deleterious effect on mechanical behavior. Little effect is noted on tensile properties at concentrations less than 500 ppm.[14] A decrease in both the strength and ductility are observed at concentrations greater than 500 ppm. However, the impact properties are severely affected by small concentrations of hydrogen. Modified Charpy, sub-size impact specimens of Zircaloy-2 and Zr-1.5 Nb which were tested after a short exposure (2 days) at 850 F in steam to introduce hydrogen exhibited an increase in the impact transition temperature[15] as shown in Figure 1. Zircaloy-2 was much more sensitive to the autoclave conditioning, having an increase in the impact transition temperature of over 200 F. The change was attributed to the presence of 69 ppm of hydrogen. Deformation under impact conditions of rapid deformation rates and triaxiality of stress occurs to

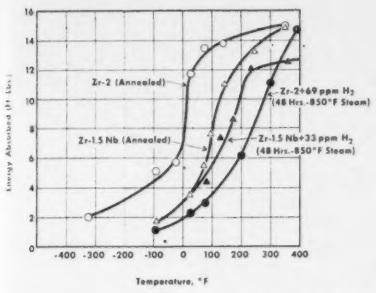

Figure 1—Effect of hydrogen pickup on impact behavior of zirconium alloys.

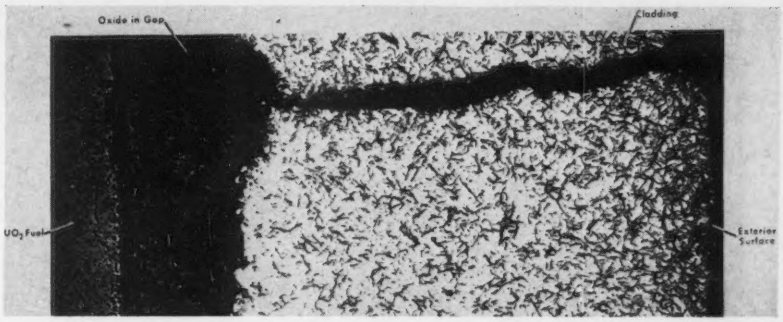

Figure 2—Hydrogen pickup during irradiation in 545 F boiling water and resultant hydride redistribution over temperature gradient. Zr-2 clad BWL fuel element exposed 485 hours.

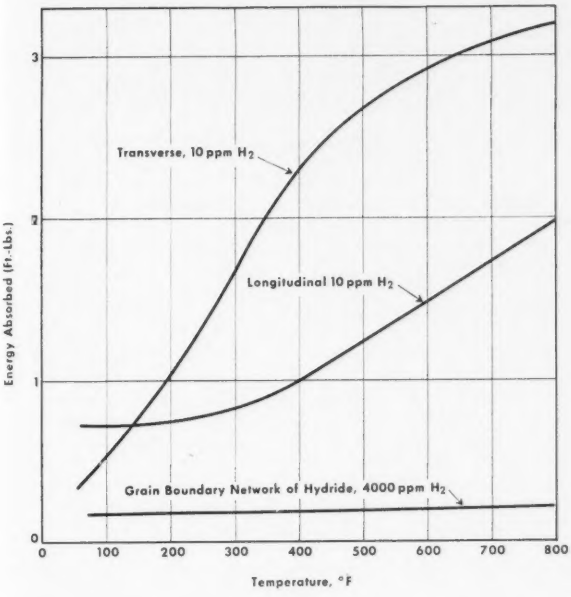
Transverse, 10 ppm H2
Longitudinal 10 ppm H2
Grain Boundary Network of Hydride, 4000 ppm H2
Energy Absorbed (Ft.-Lbs.)
Temperature, °F

Figure 3—Impact embrittlement by hydride network in cold-worked Zircaloy-2.

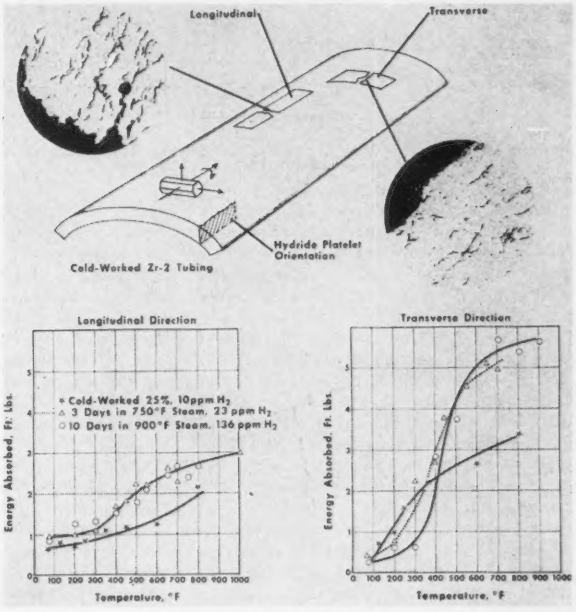

Figure 4—Effect of hydride and crystallographic orientations on impact behavior of zirconium.

a large extent by twinning. The habit plane of zirconium hydride has been shown to be the twin planes,[16] hence it is not surprising that impact properties should be so highly sensitive to hydrogen content. The lower sensitivity of the Zr-1.5Nb alloy to hydrogen may be due to the higher solubility for hydrogen[17] than in Zircaloy-2 and the lesser amount of hydrogen present, 33 ppm, for a given autoclave exposure.

The significance of hydride platelets which result from hydrogen pickup can be shown by correlations which exist between fuel element failures and hydride precipitate. It is not certain that the hydride results in the fuel element failures, although Lustman et al[18] have summarized graphically the time to failure for defected elements as a function of the heat flux. The curve divides the plot into two regions, one in which hydride failure occurs, and one in which no failures were observed. There is, however, some ambiguity as to the exact meaning of hydride failure. An example of excessive hydride precipitate in the region of a fuel element failure is shown in Figure 2,[19] although failure is not definitely related to the presence of the hydrides. Extensive hydriding can be seen; thermal diffusivity has resulted in a higher concentration of hydride at the cooler, external surface.

Quantitatively, it has been shown that a complete grain boundary network of hydride causes failure under impact at temperatures up to at least 800 F.[20] Figure 3 illustrates the effect of 4000 ppm of hydrogen which was introduced by steam autoclaving at 900 F on unetched samples of Zircaloy-2. The excessive hydrogen pickup was the result of surface preparation and does not generally occur to this extent under the conditions employed. Although hydrogen contents of this magnitude would not be expected during normal operation, it is possible to have localized concentrations of over 1000 ppm due to localized corrosion and thermal redistribution. The severe embrittlement to be expected in these regions is readily apparent. Temperature gradients and concomitant stresses arising from heat transfer conditions at high fluxes serve to magnify the problem.

Although brittleness is associated with precipitated hydrides, there are other factors which contribute to brittleness.[20] Figure 4 shows the influence of crystallographic orientation, hydride platelet orientation, and cold work. Consider first cold-worked Zircaloy-2 containing 10 ppm hydrogen. Samples cut in the transverse direction require less energy absorption for fracture than longitudinal samples. The orientation of the basal planes is parallel to the rolling direction, thus the transverse sample fractured parallel to these planes, and the longitudinal sample fractured perpendicular to the basal planes. At higher temperatures, however, the transverse samples required more energy for fracture than the longitudinal samples.

Now, consider the transverse samples only. Autoclave exposure caused hydrogen pickup but also permitted some recovery of the cold work to occur. The recovered samples, containing higher hydrogen, up to 136 ppm compared to 10 ppm for the cold-worked specimens,

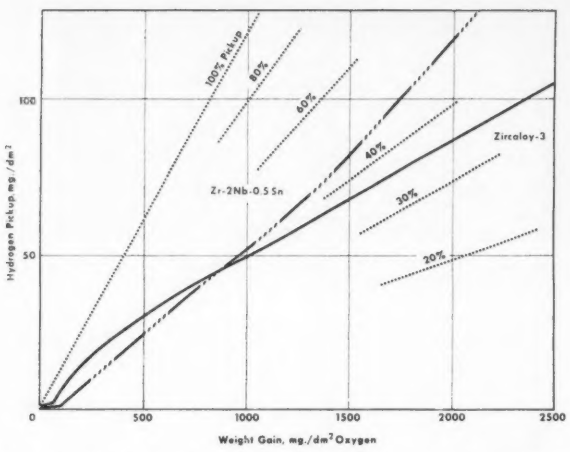

Figure 5—Dependence of hydrogen pickup on total amount of corrosion (500 C, 15 psi steam). Taken from Cox.[21]

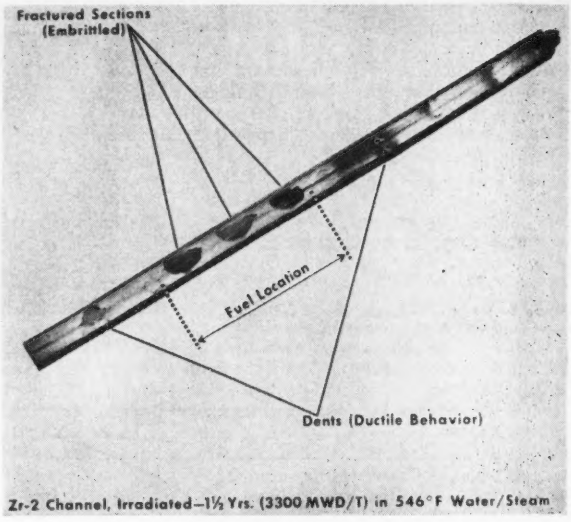
Fractured Sections (Embrittled)
Fuel Location
Dents (Ductile Behavior)
Zr-2 Channel, Irradiated—1½ Yrs. (3300 MWD/T) in 546°F Water/Steam

Figure 6—Effect of irradiation and hydrogen pickup on brittle behavior during "pipe-drop" testing.

exhibited a higher impact energy by a factor of two at temperatures above 500 F. Finally, consider the hydride orientation. The platelets lay parallel to the rolling (longitudinal) direction and were predominantly in grain boundaries. The transverse direction showed a marked tendency to fracture with less energy absorption than the longitudinal samples at low temperatures. The hydride platelets were parallel to the fracture path and provided a notch effect. On the other hand, the hydride precipitate was perpendicular to the fracture path in the longitudinal samples and therefore required more energy for fracture.

The net result of this discussion is to show that high hydrogen in itself may not be too harmful if other factors are favorable, and that brittleness may occur with low hydrogen when crystallographic orientation and cold work are unfavorable.

The rate of hydrogen pickup is not generally cited in the literature. Rather, the pickup fraction or, in some cases, the weight gain of hydrogen per unit area is reported. Two or three pieces of datum are given, and it is assumed that the pickup fraction is constant during the different stages of corrosion. Recent work by Cox[21] has shown a pronounced variation in pickup rate as the corrosion process goes through the interference color, parabolic, and linear stages. Cox reports for Zircaloy-2 at temperatures ranging from 350 to 600 C in 1 atm steam values of 30-50, about 10-15, and 80-100 percent pickup, respectively, for the three stages. These trends are more apparent when the hydrogen uptake is plotted against the weight gain of oxygen. Distinct portions of the plot are observed, and the behavior is not uniform as generally assumed by most investigators. If the corrosion is not carried past transition at low temperatures, a value for the hydrogen pickup is obtained which is much too low for the rate of pickup after transition. Cox has corroded several alloys for periods of time in excess of transition and observed crossing of the hydrogen pickup versus oxygen weight-gain curves. This behavior is illustrated in Figure 5. Although Zircaloy-4 appears to have a lower pickup fraction of hydrogen than Zircaloy-2, caution should be exercised insofar as complete acceptance of Zircaloy-4 is concerned for use beyond transition on the basis of available hydrogen pickup data. The rate of pickup should be examined in detail at high weight gains, and then a valid comparison can be made.

The combined effect of irradiation and hydrogen on ductility has not been examined in detail. A limited amount of data is available and shows that both effects together are much worse than either alone. A pertinent experience with irradiated Zircaloy-2 channels in VBWR sheds valuable qualitative information on this problem. Ogawa and Brandt[22] report that certain channels which performed well during irradiation exhibited failures of small holding tabs during subsequent out-of-pile handling. The channels were therefore removed and stored in a fuel storage pool. The channels were to be scrapped, and in an effort to reduce the storage space, the channels were collapsed to a smaller volume by striking them with a heavy pipe under water. Those portions which had been across from the fuel failed in a completely brittle manner when the 57-pound weight was dropped from a height of 12 inches. Areas above and below the fuel did not fail during the "pipe-drop" test but merely deformed plastically. Figure 6 shows the holes and dented areas in one such channel. Further examination revealed the presence of 45 ppm of hydrogen in the irradiated area and about 20-82 ppm of hydrogen in the ductile portions. Most of the brittleness may be attributed to the radiation damage, but it is felt that the brittleness associated with a given amount of hydrogen is much greater when subjected to irradiation (in this case 7.5×10^{20} nvt thermal and 8.9×10^{20} nvt epithermal and fast.)

Columbium

As stated earlier the use of columbium alloys can be justified only for unusual operating conditions in view of the prohibitive cost of mill products. Two examples would be for superheat steam reactors and for liquid metal-cooled reactors. The high temperatures of superheat reactors rule out the use of zirconium alloys, and the incentive to use columbium over stainless steel lies in the lower cross section of columbium. Insofar as liquid metal reactors are concerned, columbium alloys are very promising with respect to both high strength and excellent corrosion resistance to most molten metals.

Screening tests of numerous columbium alloys in 750 F steam revealed many alloys which possessed good corrosion resistance.[23] Some Cb-V binary alloys exhibited a good combination of properties including corrosion resistance to 750 F steam and were therefore studied in greater detail. An intensive study of Cb-V binary alloys[24] showed that protective films formed at 900 F; the corrosion process followed the cubic rate law for exposures at least to 49 days. Unpublished research by Douglass[25] has shown that a Cb-8.9 a/o V alloy followed the parabolic law in superheat steam, 1050 F, up to at least 3000 hours. These data represent the extent of corrosion tests performed in high temperature steam. On the basis of these preliminary results it appears likely that adequate corrosion resistance may be attained. The effect of higher temperatures up to 1300 F has not been studied.

Corrosion Behavior

The effect of temperature on the corrosion rate in steam is shown in Figure 7. The transition phenomena observed in most zirconium alloys have not been seen in Cb-V binary alloys containing greater than about 7 a/o vanadium. An alloy containing 12.6 a/o V did not undergo transition in 7100 hours in 750

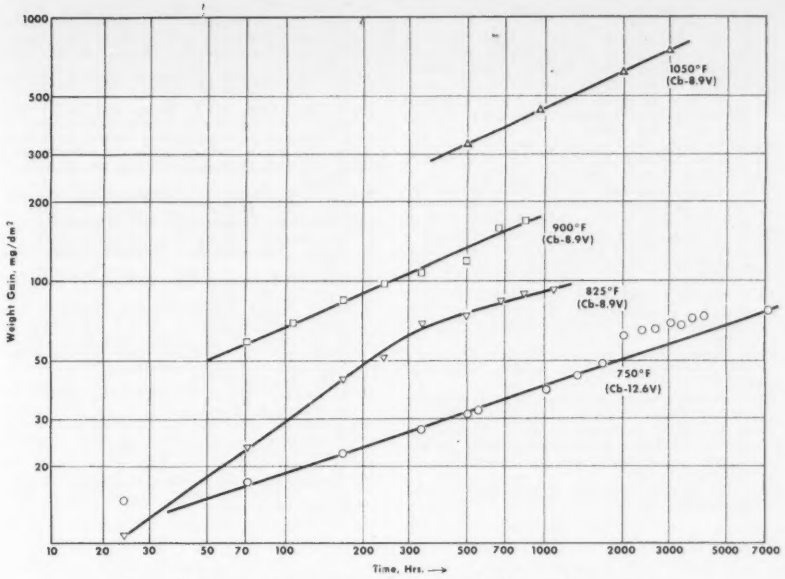

Figure 7—Corrosion behavior of Cb-V Alloys in high temperature steam.

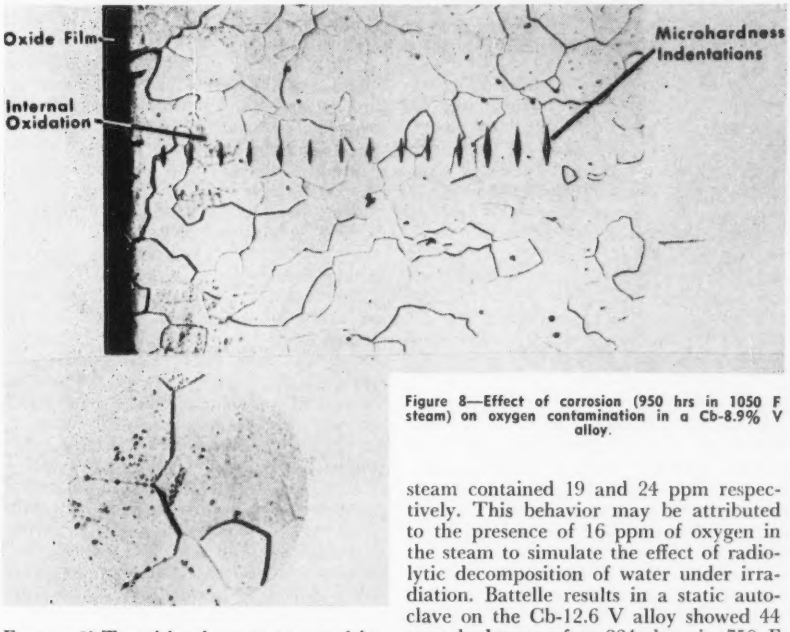

Figure 8—Effect of corrosion (950 hrs in 1050 F steam) on oxygen contamination in a Cb-8.9% V alloy.

F steam.[23] Transition has not occurred in duplicate samples of a Cb-8.9 a/o V alloy in 3000 hours in 1050 F steam in a dynamic loop test.[25] Other columbium alloys spalled at shorter times, and lost weight rapidly. If weight gains were to be calculated from metal loss measurements, it is apparent that the parabolic to linear change would be observed; however, columbium alloys do not maintain dark protective films when the rate law changes. Spalling definitely occurs and is associated with weight losses.

Hydrogen pickup does not present a problem in the corrosion resistant alloys. The amount of hydrogen picked up is nil according to the unpublished results of G. E. loop tests. The Cb-8.9 V alloy had an initial hydrogen content of 16 ppm, and after 500 and 950 hours in 1050 F steam contained 19 and 24 ppm respectively. This behavior may be attributed to the presence of 16 ppm of oxygen in the steam to simulate the effect of radiolytic decomposition of water under irradiation. Battelle results in a static autoclave on the Cb-12.6 V alloy showed 44 ppm hydrogen after 224 days in 750 F steam.[26]

A different type of problem from those encountered in zirconium alloys must be considered in columbium alloys for high temperature operations. Oxygen contamination becomes appreciable because the temperature is high enough for oxygen to diffuse into the metal from the oxide-metal interface. Zirconium alloys act as a sink for oxygen, but because of the low temperature and negligible diffusivity of oxygen at temperature, the oxygen-contaminated zone is restricted to a very narrow layer of metal. The effect of contamination on the Cb-8.9 V alloys is shown in Figure 8 on a taper section for a sample corroded 1000 hours in 1050 F steam.[25] Some internal oxidation took place as may be seen by the finely dispersed oxide particles. Further evidence of contamination can be seen from the hardness indentations. The depth of contamination is much greater than in any zirconium alloy, although similar data for zirconium alloys are lacking at this temperature. Unless a barrier is provided, sufficient oxygen contamination can occur to embrittle the alloy within reasonable periods of time.

Various possibilities exist to circumvent the preceding problem. Alloying additions may be made which do not impair the corrosion resistance of the alloy but serve to decrease the diffusivity of oxygen. Sims, Klopp, and Jaffee studied the contamination of several binary columbium alloys during air oxidation[27] and noted that an increase in the titanium content from 1 a/o to 25 a/o reduced the contamination rate constant and diffusion coefficient of oxygen by a factor of about 50 over the temperature range studied. Likewise, the effect of zirconium was more pronounced in reducing the contamination constant and depth of hardening. The use of alloying additions to reduce the depth of hardening by reducing the diffusivity of oxygen in the alloy is predicated upon their effect on corrosion resistance, fabricability, etc. Much work needs to be done in this area.

Another alternative is the use of a protective coating such as the zinc coatings investigated by Sandoz.[28] It has been shown that these coatings were self-healing during oxidation in air from about 1300 to 2200 F and effectively eliminate oxygen contamination in the base metal. It remains to be seen, however, how these coatings would behave in superheat steam.

Liquid Metal Applications

A great deal of interest has arisen recently over the use of columbium in liquid metal-cooled reactors. The high temperature strength, relatively low cross section, and good corrosion resistance to liquid metals make columbium very attractive as a cladding and structural material for nuclear turboelectric systems in space power systems.[29] Efficient operation and low weight require high operating temperatures and high heat fluxes. A final, but extremely important criterion is virtually nil corrosion and mass transfer in these systems which remain unattended for periods in excess of one year.

The chief problem with respect to sodium systems is the marked sensitivity of columbium to small amounts of oxygen in the system.[30] The corrosion rate becomes quite high in the presence of oxygen due to the strong reducing power of sodium which does not permit a protective oxide film to form. Instead, removal of columbium proceeds, the final product being $(Na_2O)_4Nb_2O_5$. If sufficient precautions are taken in the form of hot 'trapping," the corrosion resistance of columbium is excellent.

Insufficient data are available to generalize on the corrosion resistance of concomitant problems in the presence of other liquid metals. Much work is presently getting under way and should be available within the next year.

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Any discussion of this article not published above will appear in June, 1962 issue

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DISCUSSIONS

Corrosion of Reinforcing Steel in Concrete in Marine Atmospheres by Howard F. Finley. Corrosion, Vol. 17, No. 3, 104t-108t (1961) Mar.

Comments by R. F. Stratfull, Corrosion Engineer, California Division of Highways Materials and Research Department, Sacramento, California.

The advanced degree of deterioration on the 26-year-old Lake Maracaibo platform was viewed with considerable interest. Its advanced deterioration again points out that corrosion of reinforcing steel can be an economic factor in the use of reinforced concrete structures in corrosive environments.

The author points out an instance where reinforcing steel has corroded when the environment was apparently free of chlorides. In Shermer's report[1] of this corrosion in the apparently chloride free environment, the only data reported were that the concrete had a high water cement ratio and was porous, with low strength, and that the concrete cover over the steel was less than one inch. Also, it appears that this concrete which had a reported strength of 3000 to 4000 psi after 8 years was exceedingly poor to begin with or had deteriorated in strength since its construction.

It is common knowledge that concrete can be deteriorated by sulfates, acids, carbon dioxide and a host of other environmental constitutents. To this writer's knowledge, there have been no known reports of the corrosion of concrete embedded steel unless one or two factors are present. These factors are: (1) the deterioration of the concrete, or (2) the contamination of the concrete. Of course, stray or impressed currents have been shown to cause corrosion in non-deteriorated or contaminated concrete.

It can be said that the constituents in concrete chemically inhibit the corrosion of steel while the mass resists the physical entry of contaminants. Therefore, we can speculate that as long as the concrete remains unchanged, so does the steel. An illustration of this opinion is shown by the work of Tyler[2] which indicates that the corrosion of steel is less in a concrete that has the greater resistance to physical deterioration by the environment.

When chlorides of sufficient quantity enter concrete and progress to the surface of the steel, it appears that the mechanism of corrosion thereafter cannot be attributed to one factor or cause. For instance, in previous work it was shown[3] that sea salts reduced the pH of a cement extract solution, destroyed the passive condition of the steel and changed the half cell potential of steel from a passive to an active value. This work also showed that when a concrete block was cast with differentials in sea salt content, the steel in the concrete with the higher salt content was anodic.

Observations of the half cell potentials of steel in solutions containing a large quantity of added sea salts indicated that sufficient voltage may not result and cause corrosion by differentials in salt content, providing that the concentration of sea salts is sufficiently great. In this case (of high salt content) differentials in the moisture content of a concrete could be the predominant cause of the corrosion. Of course, this does not mean that, when concrete contains a large quantity of sea salts, the steel would not corrode in a manner similar to that observed in submerged or atmospherically exposed steel. In this case it is assumed that a continued rate of corrosion would depend upon the rate of the diffusion of oxygen through the moisture contained in the concrete to the surface of the steel.

It was with interest that the work of Schaschl and Marsh[4] was reviewed for possible application to the corrosion of reinforcing steel. In a solution of pH 7 they found that the electrode immersed in the solution of greater salt content would be anodic to one in the solution of lesser salt content. They also brought out that the anodic condition was caused by a difference in oxygen concentration between the solutions of different salt content. The difference in oxygen concentration was attributed to the difference in solubility of oxygen in the two different salt solutions. Therefore, it is apparent that when other conditions are equal, a differential aeration cell would be the result of a differential in salt content. There are other causes of differentials in oxygen concentration, but in this discussion we are only considering salt content and its influence on the differential oxygen cell. One other factor that should be considered about the work of Schaschl and Marsh is that their experiments did not involve solutions that contained a pH of 12.5, which is the approximate pH of a saturated cement extract solution, nor did they combine solutions of say 12.5 and 7.0 and then vary the salt content to determine the effect of chlorides upon corrosion of the steel.

If we consider that differentials in oxygen concentration of themselves constitute the cause of corrosion of concrete embedded steel, then we would expect to find corrosion in almost any environment. For instance, the corrosion of steel in the concrete pile of a structure, a portion of which is exposed to fresh water and to air, should be as common as that observed for a pile exposed to sea or other aggressive waters. However, this has not been the case. At least, the reports of such occurrences are decidedly lacking in the literature. The reports of corrosion in non-marine environments have generally been concerned with instances where there has been leaching of the soluble constituents of the concrete or other physical or chemical reactions of the environment to the concrete and has resulted in the corrosion of the steel.

References

1. C. L. Shermer. *Civil Engineering*, 26, 56-57 (1956) Dec.
2. I. L. Tyler. Long Time Study of Cement Performance in Concrete. *Journal of the A.C.I.*, V. 31, No. 9 (1960) Mar. Also see Discussion by R. F. Stratfull. *J.A.C.I.* 32, No. 3, Part 2, (1960) Sept.
3. Bailey Tremper, J. L. Beaton, R. F. Stratfull. Causes and Repair of Deterioration to a California Bridge Due To Corrosion of Reinforcing Steel in a Marine Environment, Part II, Fundamental Factors Causing Corrosion. Presented at the 36th Annual Meeting. Highway Research Board Jan. 7-11, 1957, HRB No. 182.
4. Edw. Schaschl, G. A. Marsh. Concentration Cells and Aqueous Corrosion. *Corrosion*, Vol. 16, No. 9, 461t-467t (1960) Sept.

Reply by Howard F. Finley:

The comments by Stratfull were studied with considerable interest. His principal point was that to his knowledge there have been no known reports of the corrosion of concrete embedded steel unless there has been deterioration or contamination of the concrete. The writer agrees that when either has occurred that it is almost a certainty that corrosion of the reinforcing steel will occur. But this simply means that conditions are mutually favorable for these three independent phenomena. The corrosion accompanies the deterioration and contamination without being caused by them. The necessary and sufficient conditions for corrosion of reinforcing steel in concrete are the presence of liquid water and a continuing supply of oxygen. Favorable conditions exist in porous, permeable concrete.

Although there are several reports in the literature of corroded reinforcing steel in controlled laboratory experiments where there was no chance for deterioration or contamination of the concrete, a single example will suffice to illustrate the point.[1]

It may seem an exercise in semantics to labor the point of whether corrosion of reinforcing steel in concrete is caused by differential aeration cells produced by differing salt concentrations or whether it is caused by salt contaminated concrete; that is, until one important economic consideration is introduced.

Can concrete be batched with brackish or salt water? In many areas of the world such as the Lake Maracaibo area fresh water is at a premium and brackish water is used in concrete construction. Creole Petroleum Corporation has manufactured and driven more than 30,000 concrete piles in Lake Maracaibo in water depths from 15 to 110 feet. All the concrete was mixed with Lake Maracaibo water whose salinity varied from 750 to 3600 ppm. The course aggregate was mined but washed with ocean water and the fine aggregate was dredged from the lake bottom. All of this concrete is con-

taminated with salt but none of the piles has the typical reinforcing steel corrosion. In marked contrast are many platform caps and beams that were poured in place. High water-cement ratios were used for easier placement under difficult working conditions. Many of these caps and beams such as those in the 26-year-old platform have corroded reinforcing steel. They are made with the same cement and aggregate and are exposed to the same environment as the piles. The only real differences between them and the piles are water cement ratio, porosity, and permeability. Actually the piles, being closer to the water, are in a more favorable position to absorb lake salts and result in corrosion if the salt contamination mechanism is the correct one.

Stratfull notes that if differential oxygen concentration is the basic corrosion mechanism then it would be expected that corrosion of reinforcing steel would be found more often in inland areas where it is not frequently found. The fact that such is not the case probably results from the less severe environment. For example, the humidity requirements for corrosion are probably quite strict and are met only near large bodies of water. Then too, when porous permeable concrete favorable to corrosion of reinforcing steel is located alongside a body of salt water it will accumulate sea salts which will accelerate the corrosion.

Reference

1. D. H. Pletta, E. F. Massie, and H. S. Robins, *Journal, Am. Concrete Inst.*, 21, No. 9, 513-525 (1950) March.

Kinetics and Mechanism of Hydrogen Attack of Steel by L. C. Weiner. Corrosion, Vol. 17, No. 3, 137t-143t (1961) Mar.

Discussion by K. Haefner, Research and Development Department, American Oil Company, Whiting, Indiana.

Dr. Weiner is to be congratulated on his systematic approach to the complex problem of hydrogen attack of steel. However, two fundamental difficulties seem inherent in his treatment.

Equation 3 indicates that incubation time is a continuous function of exposure temperature and pressure at constant grain size and constant carbide shape below 1000 F. The Nelson curve, giving operating limits for low-carbon steel in hydrogen service, on the other hand, shows hydrogen attack (t_o = finite time) as a discontinuous function of temperature and pressure. For example, at 500 F or 533 K and 800 psi, Equation 3 gives an incubation time of 2754 hours, whereas the Nelson curve indicates that no attack should occur.

Despite the proposed mechanism governing the incubation period, the occurrence of iron oxides at grain boundaries is rare in steels containing C, Mn, Si, and Al. Moreover, the possibility of CO diffusion in iron is still under dispute. One should therefore proceed with caution.

Reply by L. C. Weiner:

Mr. Haefner's comments are appreciated, but the author cannot agree with him in regard to fundamental difficulties being inherent in the study.

Although it is true that the Nelson curve as drawn shows hydrogen attack as a discontinuous function of exposure temperature and pressure while the present study indicates a continuous function of attack up to 1000 F (all steel variables held constant), there appear to be no sound reasons to expect such a discontinuous function based on either the mechanism that has been proposed or any other physical model that has been suggested.

If in reality the Nelson curve was truly discontinuous rather than asymptotical to the ordinate, consideration must be given to the difference in exposure conditions employed in this study relative to those on which the Nelson curve is based. While the steel used in this study was exposed on all sides to hydrogen at equilibrium temperature and pressure, the data from which the Nelson curve has been established in general involved steel exposed from only one side and certainly not under equilibrium nor quasi-equilibrium conditions. The latter will result in non-uniformity of attack which has been repeatedly observed as well as creating uncertainty and scatter in temperature and pressure data.

Because of the make-up and factors on which the Nelson curve is based, it is the opinion of the author that caution must be exercised in utilizing it. It must be remembered that the Nelson curve is *not* based on constant chemical composition, grain size and other steel variables that may affect attack, nor does it take into consideration the effect of exposure time, an extremely important variable in hydrogen attack as has been aptly demonstrated. In addition, the actual data points that are shown to be "safe" may in fact not be so. Failure to observe hydrogen attack in steel structures in service could stem from many reasons, some of the more pertinent ones being: (a) insufficient load on the structure to cause failure; (b) if test samples were taken, the non-uniformity of attack in the areas from which they were removed; (c) insensitive methods of detecting attack. Lastly, because of the sparsity of data points, uncertainty in exposure temperature and pressure data and non-uniformity of steel variables, it is felt that the Nelson curve should not be drawn as a definitive line separating "safe" from "unsafe" exposure conditions; a band would seem more appropriate. Thus, on the basis of this analysis of the Nelson curve, the results of this study and proposed mechanisms of attack, it is the opinion of the author that in reality this curve asymptotically approaches the ordinate and the discontinuous nature as it is drawn is but an artifact.

The example that Mr. Haefner has cited in his discussion, 500 F and 800 psi, vividly points out the care that must be used in employing the Nelson curve. At this set of exposure conditions, the Nelson curve predicts freedom from hydrogen attack for an unspecified period of operation—which could be interpreted as infinity, since this curve cannot take into consideration exposure time. However, close examination of the curve reveals that this exposure point falls just below the drawn line, in fact, just about 10 F below it. Certainly the Nelson curve is not so accurate and precise to give safe limits within ±10 F. More instructional and of greater use than the Nelson curve would be a series of T_E vs P_E curves for various values of incubation times, t_o, and of course, for various compositions, grain sizes and other steel variables. Such curves can be determined from relationships as described in Equation 3.

With respect to the second part of Mr. Haefner's discussion, the author agrees that one would not expect large amounts of iron oxides at grain boundaries in steels containing C, Mn, Si and Al, even though there is a definite amount of dissolved oxygen in equilibrium with these constituents. However, when monomolecular layers are concerned, the picture is much different. Calculations show that for the steel studied (ASTM grain size #7.5), only 0.0013 percent by weight of oxygen is needed to form a continuous monomolecular layer of iron oxides at *all* grain boundaries. This percentage is but a quarter of that determined in this steel. Even assuming that half of the total oxygen content in this steel is tied up as oxides of the other constituents, there is ample oxygen in the steel to form at least a monomolecular layer of iron oxides at all the grain boundaries.

It is true then that the presence of these constituents in the steel will limit the amount of grain boundary iron oxides and hydrogen attack will occur in a relatively short period of time as was experimentally demonstrated. Any factor that decreases the amount of grain boundary iron oxides (i.e. decreasing grain diameter), decreases incubation time for attack. This is exactly the behavior predicted by the proposed mechanism and experimentally demonstrated with the iron alloys, grain size study and killed vs semi-killed steel study in this investigation. Of great value would be an experimentally determined quantitative relationship between grain boundary iron oxides content and incubation time.

In regard to the diffusion of CO in iron, it is hoped that the doubt shrouding this subject will soon be dispelled now that more advanced methods of diffusion measurements are available.

Corrosion of Superalloys at High Temperatures in the Presence of Contaminating Salts by A. Moskowitz and L. Redmerski. Corrosion, Vol. 17, No. 6, 305t-312t (1961) June.

Question by Robert D. Eberhardt, 3508 N. 61, Milwaukee 16, Wisconsin:

It was noticed that all alloys were stressed to the same level regardless of

their ultimate tensile or yield strength. Would you expect any difference in the order of performance if each alloy had been stressed to an equal percentage of its yield strength at the test temperature?

Reply by A. Moskowitz:

It should be noted that the tests were performed at high temperatures under creep-testing conditions, rather than under the low temperature conditions wherein materials are frequently stressed to equal percentages of yield strength. There need not be a correlation between the relative yield strengths of different alloys at a given elevated temperature and their relative creep-rupture strengths at that temperature.

Available data on the 0.2 percent offset yield strengths for the tested alloys are listed in Table 1.

TABLE 1—Offset Yield Strengths for Tested Alloys

Alloy	1600 F	1700 F	1800 F
WF-11.....	34,000 psi	29,000	24,000
M-252.....	58,000	.....	.....
Rene 41.....	72,000	47,000	30,000
Inconel X.....	Approx. 32,000	.....	.....
Inco 702.....	Approx. 32,000	.....	.....

So for data involving stressing to equal percentages of yield strengths at 1600 F, the results for WF-11, Inco 702, and Inconel X at a given stress (5 ksi) could be compared to the results for the M-252 and Rene 41 at approximately double the stress (10 ksi). The stress differences would be less at the higher temperatures. The WF-11 might then rank in some instances above the M-252 and Rene 41, but major differences in rankings would not be produced within the 30-hour test time limitation.

It is interesting to choose stress conditions for each alloy at a given temperature such that creep-rupture occurs in similar times without salt present ("equal creep-rupture" conditions), and then tabulate times to failure for these conditions with salt contamination. Examination of the data indicates that the times to failure for the different alloys when salt contaminated are similar under these "equal creep-rupture" conditions. This correlates with the conclusion that all the alloys are similarly susceptible to the salt corrosion.

Effect of Mineral Impurities in Water on the Corrosion of Aluminum and Steel by L. C. Rowe and M. S. Walker. Corrosion, Vol. 17, No. 7, 353t-356t (1961) July.

Comments by H. P. Godard, Aluminium Laboratories Limited, Kingston, Ontario, Canada.

The authors are to be congratulated on a major contribution to the literature on the influence of water composition on the corrosion behavior of aluminum and steel. Their strip conductance technique has the advantage of speed and will undoubtedly be used by others in further studies of this kind.

The results obtained with aluminum agree well with those reported by others and with our own experience. I am familiar with only one further paper in the field, by Sverepa.[1]

I would ask for a more complete description of the distilled water used as a base to prepare the various test solutions. Specifically, what was its resistivity and copper content? Water from commercial stills is inadequate for work of this kind and should be redistilled in glass to remove completely the last traces of minerals and particularly copper ion. The resistivity should be at least 109,000 ohm cm.

Although it was found that 2 ppm copper in aerated distilled water did not increase the amount of corrosion, I am familiar with two cases where 0.10 ppm copper in distilled water led to extensive pitting of aluminum. This suggests that there may be a critical low range of copper content above which no pitting occurs. One experiment we have made supports this suggestion.

While the authors state that copper is not a normal impurity in municipal waters, our analysis of one hundred natural waters has shown a range of copper content from less than 0.001 ppm to 0.30 ppm with most containing less than 0.010 ppm. Porter and Hadden[2] have reported that 0.020 ppm is sufficient to influence the behavior of aluminum.

We have also observed that pits initiate at the site of gas bubbles adhering to the surface. It may be of interest to note that if these are removed over a period of several hours with a camel hair brush, pits do not initiate.

Other information on the influence of temperature on the pitting of aluminum has been reported by Godard.[3] By the use of an individual pit current technique it was found that in some pits activity reached a maximum at about 40 C while with others activity decreased as the temperature was raised.

Although the work to date has revealed a number of trends, it is still not possible to predict with certainty the behavior of aluminum from a table of water analyses. Much more work remains to be done.

References

1. O. Sverepa. Corrosion of Aluminium and Its Alloys in Waters of Different Composition. Werkstoffe und Korrosion, 9, 533-536 (1958).
2. F. C. Porter, S. E. Hadden. Corrosion of Aluminum in Supply Waters. *J. Appl. Chem.*, 3, 385-409 (1953).
3. H. P. Godard. The Corrosion Behaviour of Aluminium in Natural Waters. *Can. J. Chem. Eng.*, 38, 167-173 (1960).

Reply by Leonard C. Rowe:

Comments by Dr. Godard are always appreciated. His remarks regarding the distilled water used in the tests are certainly appropriate, although our distilled water apparently satisfied his minimum requirements. The copper content was of the order of three parts per billion, and the resistivity was in the range of 400,000 to 500,000 ohm cm.

Although our experience did not show a significant effect from copper as a single impurity in water, it is possible that it might produce pitting under other conditions or with the use of aluminum having a composition different from that used in the tests. The time of exposure can in itself be a significant factor. Since Dr. Godard did not discuss these variations, we must defer to his judgment on this matter.

Our remarks concerning the presence of copper as a normal impurity in water were made with regard to a significant amount being present. Dr. Godard appears to bear out this fact when he states that most waters that they have analyzed contained less than 0.010 ppm of copper.

Effect of Sunlight on the Corrosion of Steel (Topic of the Month) by Leonard C. Rowe. Corrosion, Vol. 17, No. 6, 267t-268t (1961) June.

Comments by B. F. Brown:

Some time ago several hundred specimens of various alloy white cast irons were stored in a laboratory for a total of about eighteen months. The available artificial illumination was minimal, and most of the daytime illumination came from a window about 15 feet away from the specimen holders. At no time of the year did direct rays of the sun strike the specimens. At the end of the eighteen months, all the surfaces of the specimens which could "see" the windows were covered with a light red rust, which was absent from those areas which did *not* "see" the window. The effect was striking, with many clear-cut examples of the shadow of one specimen affecting the corrosion rate of a neighboring specimen. This was taken to indicate that wave lengths other than ultraviolet were promoting atmospheric corrosion. The dark sides of specimens did not corrode as rapidly as illuminated surfaces more remote from the window, which was taken to indicate that the effect was not to be attributed to atmospheric contamination entering from the window.

Reply by Leonard C. Rowe:

The comments by Mr. Brown are appreciated. He has mentioned some anomalous effects which associate corrosion with light exposure. Similar effects have been noted by other individuals, and brief comments concerning these experiences have appeared in the literature.

From an energy concept, one would expect ultraviolet light to have a greater effect on this phenomenon than light of other wave lengths. It is difficult to isolate the effect of light, because other factors are necessary for corrosion and usually make a greater contribution. Light passing through a window glass is filtered to a degree, depending upon the

thickness and type of glass, and it is difficult to determine which portion of the light contributes to the corrosion.

Stress-Corrosion Cracking: A Review of Current Status (An Educational Lecture), by W. Lee Williams. Corrosion, Vol. 17, 340t-344t (1961) July.

Comments by F. A. Champion, The British Aluminium Company Ltd., Chalfont Park, Gerrards Cross, Bucks, England:

It has always seemed to me that for a definition to serve a useful purpose, it is desirable for it to be definite and simple. In this country the following definition of stress corrosion is generally accepted.* "The term 'stress corrosion' implies a greater deterioration in the mechanical properties of the material through the simultaneous action of a static stress and exposure to corrosive environment than would occur by the separate, but additive, action of these agencies."

Such alien factors as corrosion fatigue and diffusion of liquid metals are excluded by this definition. The adoption in the U.S.A. of two definitions, namely, "stress corrosion" and "stress corrosion cracking" was understandable, although the "definitions" seemed rather vague. The undesirability of the latter feature has now been demonstrated by the wonderful list of factors included under these two umbrellas by Williams, and apparently earlier by Copson. This seems to me to offer full scope for confusion, rather than a promise of a better understanding of the behavior involved.

Reply by W. Lee Williams:

I wish to thank Mr. Champion for his interesting and thought-provoking discussion. His comment is most welcome, and it points to the need for engineers (and scientists) to standardize their terminology. Indeed, I was hopeful that my lecture would prompt this type of discussion.

The opening sentence of my lecture recognized the lack of universally accepted definitions, and much of the text was devoted to definition and explanation of terms. The intent was not to confuse the subject by making it complex, but to make the subject perfectly clear through recognition and classification of the complexities. Also, the intent was not to propose my own set of definitions as standard, but to insure that everyone understood what was meant within the confines of my lecture.

My own belief is that the terminology associated with stress corrosion has even deeper implications. For example, how would one define the single word "corrosion"? Would diffusion of liquid metals or hydrogen embrittlement be included under a definition of corrosion? If so, one would have some justification for defining "stress corrosion" (and possibly "stress-corrosion cracking") in terms general enough to include these modes of attack when the end result is influenced by the presence of stress.

J. Inst. Metals, 1945 (Aug.) xxii, 1955, 83, 385.

The Use of Pipe-To-Soil Potential in Analyzing Underground Corrosion Problems by Bernard Husock. Corrosion, Vol. 17, No. 8, 391t-395t (1961) Aug.

Questions by Mark F. Adams, Division Industrial Research, Washington State University, Pullman, Washington:

Assume a complicated pipe network as found in city utility service. How can the potential measurements have any significance? How would the potentials be interpreted?

Reply by Bernard Husock:

It is obvious that a complicated pipe network, as found in city utility service, can result in very complicated problems and in potential measurements which are not always easy to interpret. However, it is believed that with an astute application of the statements given in the paper, potential measurements of pipe networks can be made to yield significant results. Before attempting an interpretation of the measurements, complete and accurate information concerning all of the underground structures in the area must be obtained. As stated in the paper, this is often one of the more difficult jobs for the corrosion engineer. Even if the corrosion engineer is interested in investigating only one type of utility, such as the gas lines, it is possible that all of the other utilities such as water lines constructed of cast iron, copper and galvanized pipe would have some effect on the potential of the gas lines. If the gas lines are newly installed, well coated, and insulated from the other structures, the potentials to be expected on those gas lines would be as indicated in Statement 5 under interpretation of "Potentials Under Non-Stray Current Conditions" in the paper. If unusual potentials are encountered, an investigation should be conducted to determine whether the insulators are shorted and whether the coating is as good as was expected.

The interpretation of potentials in a complicated network is not easy and certainly the paper cannot hope to cover all of the possible conditions which can be encountered in the field. The paper can be used as a guide but is not intended to be a substitute for field experience and engineering judgment.

The Sinter Plant Corrosion Problem by R. J. Schmitt. Corrosion, Vol. 17, No. 9, 425t-429t (1961) Sept.

Question by Warren E. Berry, Battelle Memorial Institute, 505 King Avenue, Columbus 1, Ohio:

In your investigation, did you consider the addition of ammonia or ammoniated compounds to the flue gas to inhibit corrosion, or was this not practical for your application?

Reply by R. J. Schmitt:

The addition of ammonia or ammoniated compounds to the exhaust gas was thoroughly studied and was found to be impractical in view of the very large volume of gas to be treated.

Question by Mark F. Adams, Division of Industrial Research, Washington State University, Pullman, Washington:

How did you handle the difference in coefficient of expansion between the steel structure and the Lumnite cement?

Reply by R. J. Schmitt:

Lumnite cement can be formulated to provide mixes with excellent insulating properties. Therefore, by applying a relatively thick coating of the proper Lumnite mix to the walls of the exhaust-gas system, the temperature at the interface of the cement and steel was sufficiently low that it was not necessary to provide for the differences in coefficient of expansion between the steel and Lumnite cement.

NATIONAL ASSOCIATION of CORROSION ENGINEERS

Economics of Chemical Plant Maintenance Painting*

A Report of NACE Technical Unit Committee T-6D On Industrial Maintenance Painting*; Prepared by Task Group T-6D-1 on Economics of Maintenance Painting**

NACE TECHNICAL COMMITTEE REPORT
Publication 61-14

Abstract

Replies from a questionnaire indicate that the cost of industrial maintenance painting is increasing and that labor-to-material costs are in a ratio of 3 to 1. Data also are given on parameters by which the average cost of maintenance painting in six industrial categories may be estimated. Field testing is the most popular method of selecting a paint and the applied cost of systems based on sandblasted surfaces is noticeably higher than for systems on hand-cleaned surfaces.

Appearance was the most frequently mentioned reason for repainting, with availability of a budget next in significance. The concensus was that intervals for repainting indoors or out should be about four years. Engineers think improved painting practices have saved their companies about 26 percent of total painting costs.

1.2.2, 5.4.5

Introduction

DATA AND conclusions in this report were derived from replies to some of the questions in a questionnaire circulated among 800 plants in heavy chemical, organic intermediate, plastics, petrochemical, pharmaceutical and metallurgical categories. Table 1 classifies the above type plants. Table 2 shows relationship of annual maintenance painting expenditure to several bases for each of these six industrial categories.

A total of 130 replies were received to this questionnaire of which some 107 contained usable data. Some conclusions can be drawn from a study of the replies which should be helpful to those concerned with industrial maintenance painting. Among the most noteworthy of these is the conclusion indicated by data in Table 3 that the annual outlay for maintenance painting is increasing. Almost twice as many companies reported higher costs as reported lower.

It was equally obvious, as indicated in Table 4 that labor-to-material cost is in a ratio of about 3 to 1 among these plants and that surface preparation costs are not much less than are costs of application of 3-coat systems.

Average Cost of Painting

Among these six categories of industrial plants the data in Table 2 show several parameters which permit assessment of total maintenance painting costs.

Respondents indicated the following percentages of three bases of estimation:

1. Of total maintenance repair outlay, 5.70 percent.
2. Of capital investment, 0.50 percent.
3. Of total painting cost, 47 percent.

These data do not indicate a significant difference among the six industrial cate-

★ Extracted from a longer report prepared by Task Group T-6D-1.

* F. Parker Helms, Union Carbide Chemicals Co., Texas City, Texas, chairman.

** S. L. Lopata, Carboline Co., St. Louis, Mo., chairman.

TABLE 1—Table of Category Indicators

The following numbers in the tables herein refer to the indicated industrial group.

Category	Industrial Group
1	Heavy Chemical
2	Plastics
3	Organic Intermediates
4	Pharmaceutical
5	Petrochemical
6	Metallurgical

TABLE 2—Relationship of Annual Maintenance Painting Expenditure to Several Bases

Industrial Category	Percent of Maintenance Repair Outlay			
	1–4	5–8	>9	Avg.
1	11	12	12	7.08
2	5	5	2	5.4
3	3	6	3	6.8
4	..	4	2	6.6
5	1	3	1	6.9
6	2	..	..	1.4
Average				5.70
	Percent Capital Investment			
	<0.5	0.5–1	>1	Avg.
1	13	5	2	0.55
2	5	1	1	0.42
3	3	1	2	0.77
4	2	1	..	0.4
5	2	2	..	0.62
6	2	..	..	0.21
Average				0.50
	Percent of Total Painting			
	<75	75–89	90–100	
1	2	5	26	
2	3	1	8	
3	..	6	5	
4	..	1	5	
5	3	1	1	
6	1	..	2	
Average	9	14	47	

TABLE 3—Characteristics of Annual Maintenance Painting Outlay

Industrial Category	Inc.	Dec.	Cycling	Constant Amt.[1]	Varies[2]
1	19	13	6	5	5
2	9	5	2	2	2
3	7	4	2	4	1
4	5	3	1	2	..
5	3	1	1	3	..
6	4	1	..	1	1
Totals	47	27	12	17	9

[1] As percentage of plant capital investment.
[2] In proportion of operating volume.

TABLE 4—Distribution of Costs of Various Maintenance Painting Activities

Industrial Category	Miscellaneous Scaffolding, Clean-Up (percent)			
	0-5	5-10	10-20	Avg.
1	1	19	9	14.6
2	2	5	5	9.7
3	1	3	9	10.0
4	1	1	5	8.7
5	..	1	6	16.0
6	1	..	2	15.0
Totals	6	29	36	12.3
	Surface Preparation (percent)			
	0-20	20-40	40-60	>60	Avg.
1	2	8	14	6	48.1
2	1	6	4	2	39.7
3	2	2	7	2	41.6
4	1	4	2	..	28.6
5	..	5	2	1	32.0
6	1	.	..	2	47.0
Totals	7	25	29	13	39.5
	Three-Coat Application (percent)				
	<20	20-39	40-59	60-80	Avg.
1	1	14	10	5	40.6
2	2	1	4	6	49.0
3	1	2	6	4	48.3
4	..	..	2	5	62.7
5	..	..	4	3	52.0
6	..	2	..	1	38.0
Totals	4	19	26	24	48.4
	Labor-to-Material Cost (percent)				
	<25	25-49	50-75	>75	Avg.
1	1	1	16	8	67.1
2	..	..	4	7	77.0
3	..	1	11	2	71.6
4	..	..	2	5	75.6
5	..	1	..	5	74.0
6	..	..	..	3	77.0
Totals	1	3	33	30	73.7

TABLE 5—Measurement of Maintenance Painting Costs

Industrial Category	Cost Estimates Based on		
	sq ft/yr	sq ft	Total $
1	9	10	25
2	2	1	14
3	4	4	7
4	1	4	3
5	..	2	4
6	..	1	3
Totals	16	22	56

TABLE 6—Materials Purchasing and Control Practices

Industrial Category	Reciprocity	Solids/ Vol	Generic Type	$/Gal	Lab Test	Field Test	Spot Analyze[1]	Specify Formula
1	7	9	23	17	15	40	5	6
2	11	7	15	9	7	20	8	9
3	3	2	10	6	3	11	1	3
4	2	2	3	3	1	5	4	3
5	4	3	4	7	4	5	..	2
6	1	..	1	3	1	1	1	1
Totals	28	23	56	45	31	82	19	24

[1] To determine if quality is consistent.

TABLE 7—Order of Preferences of Methods of Choosing a Paint System

Industrial Category	PAINT SYSTEM	
	Low Cost/ Sq Ft/Year; High Application Cost	High Cost/ Sq Ft/Year; Low Application Cost
1	31	2
2	7	1
3	12	1
4	6	0
5	6	1
6	2	0
Totals	64	5

TABLE 8—Average Applied Cost in Cents/sq ft for Labor and Materials of Paint Systems[1]

System No.	Description
1	Three-coat alkyd
2	Three-coat vinyl
3	⅛-inch asphalt mastic
4	Three-coat catalyzed epoxy

Industrial Category		HAND CLEANED System Number				SAND BLASTED System Number			
		1	2	3	4	1	2	3	4
1	Plant Painters	25	51	30	35	21	47	30	38
	Contractors	26	25	35	30	30	42	47	52
3	Plant Painters	32	44	45	50	31	37	38	42
5	Plant Painters	25	30	15	35	63	65	30	60
	Contractors	30	35	20	40	65	70	55	75
	Average Cost	28	37	29	38	42	52	40	53

(1) Data are given only when two or more answers were received.

gories as to one having higher maintenance painting costs than the others. An exception may be Category 1 (Heavy Chemical) which shows a significantly higher order of costs in the comparisons of maintenance repair outlays and percent of total painting. Category 3 (Organic Intermediates) shows highest costs as a function of capital investment, though not significantly greater than that registered by Category 5 (Petrochemical Industry).

More than twice as many plants measure painting costs in total dollars as measure then in square feet, Table 5 indicates. Only one-third as many plants use sq. ft/year as a basis of estimating costs as use total dollars.

Selection of Paints

Field testing was far ahead of the next nearest popular method of selecting paints (by generic types), data in Table 6 show. Cost in dollars per gallon was third in popularity, with laboratory testing fourth and reciprocity buying a close fifth.

However, when replying to questions about buying preferences, respondents preponderantly favor choosing a system on the basis of cost per square foot per year rather than by the applied cost. This opinion developed from replies to a question asked in such a way that the weight of the two considerations roughly approximated cost values which often may be found in alternative painting systems. See Table 7.

The fact that the preferences of the paint engineers do not match practices followed in the plants seems to indicate that improvement is needed in liaison between the engineers and plant managements.

Surface Preparation Economics

The extent to which surface preparation by sandblasting affects the applied cost of four popular paint systems is explored in Table 8. In this table the data show that the average cost among three categories of respondents who supplied significant answers was higher for sandblasted surfaces for all four systems. It is significant that plant painters in Category 3 (Organic Intermediates) were able to apply all four systems over sandblasted surfaces at a lower cost in cents/sq ft than they were the same systems over hand-cleaned surfaces. However, because the costs for hand cleaned preparation in this category are higher than the costs registered by the other two, it may be the data are askew because the sample does not represent the true situation in the whole population.

In this table an effort also was made to distinguish the differences between plant painter costs and contract painter costs. In the two categories where comparisons appear to be valid, the cost for systems applied by plant painters was less than cost of the same systems by contract painters. However it is not known whether overhead costs were included by companies supplying plant painter costs.

Appearance Factor Is Significant

Contrary to what might be expected, appearance was first choice of most respondents as a governing factor in extent of maintenance painting. Data in Table 9 show that total maintenance budget was next in significance as a controlling factor, with executive tours a poor third in importance.

A full time paint engineer is needed when the average annual cost of maintenance painting reaches $178,000, replies tabulated in Table 10 show. The annual cost basis justifying a full-time engineer in this table ranged from a low of $90,000 a year to a high of $233,000 a year. The highest estimate was in the petrochemical industry, which also showed a high parameter of costs versus two of the three bases in Table 2.

TABLE 9—Factors Governing Extent of Maintenance Painting

Industrial Category	Total Maint. Budget	Appear.	Executive Tours	Plant Output	Other
1	22	23	6	6	6
2	11	14	1	1	2
3	7	11	3	3	..
4	3	8	3	3	2
5	3	5	2	..	2
6	2	1	1	2	..
Totals	48	62	16	15	12

TABLE 10—When Full-Time Paint Engineer Justified

Industrial Category	Average Annual Cost of Maintenance Painting ($000)
1	220
2	150
3	200
4	233
5	90
Average	178

Intervals for Repainting

When estimating the common intervals that may be expected between painting and repainting, Table 11 discloses that respondents believed for outside paints the average should be 4 years and

TABLE 11—Opinions on Painting Practices

Industrial Category	Overdesign, No Paint		Average Repaint Interval Years		Paint Pipelines <2" D		Added Cost, color vs solid grey Average Percent	Paint Pipeline Insulation Paper or Felt			
								Indoors		Outdoors	
	Yes	No	Inside	Out	Yes	No		Yes	No	Yes	No
1	6	35	4.8	4.5	16	21	23	4	30	10	25
2	0	20	4.6	3.5	8	4	13	7	8	6	9
3	0	16	4.3	3.8	6	6	42	3	11	3	11
4	2	8	4.0	3.5	5	3	22	7	0	3	5
5	1	8	3.0	3.5	5	2	33	1	6	2	5
6	0	4	3.5	4.7	3	0	15	1	2	2	1
			Average				Average				
Totals	9	91	4.0	3.9	43	36	24.6	23	57	26	56

TABLE 12—Reductions in Painting Cost Achieved by Paint Engineers

Industrial Category	Percent Savings
1	32
2	20
3	30
4	28
5	20
Average	26

for inside 3.9 years. This minor difference in intervals may be the result of the tendency disclosed in Table 9 to repaint for appearances sake or when the budget permits.

More engineers favored painting pipelines of 2 inches or less diameter than thought such pipes should be left bare. Engineers also agreed more than 2 to 1 that pipeline paper or felt insulation should not be painted either indoors or outdoors.

Insofar as agreement among those supplying data is concerned, the nearest to unanimous on a single issue is found in Table 11 where the vote against exposing steel bare without paint was 10 to 1. In this same tabulation, the opinions on the price differential between a color system and a solid grey system ranged from 13 to 42 percent, with an average added cost differential for all respondents of 24.6 percent.

Painting engineers estimated they were able to save an average of 26 percent by proper methods as compared to costs of like operations before improved methods were introduced. Table 12 gives the detailed breakdown.

Any discussion of this article not published above will appear in June, 1962 issue

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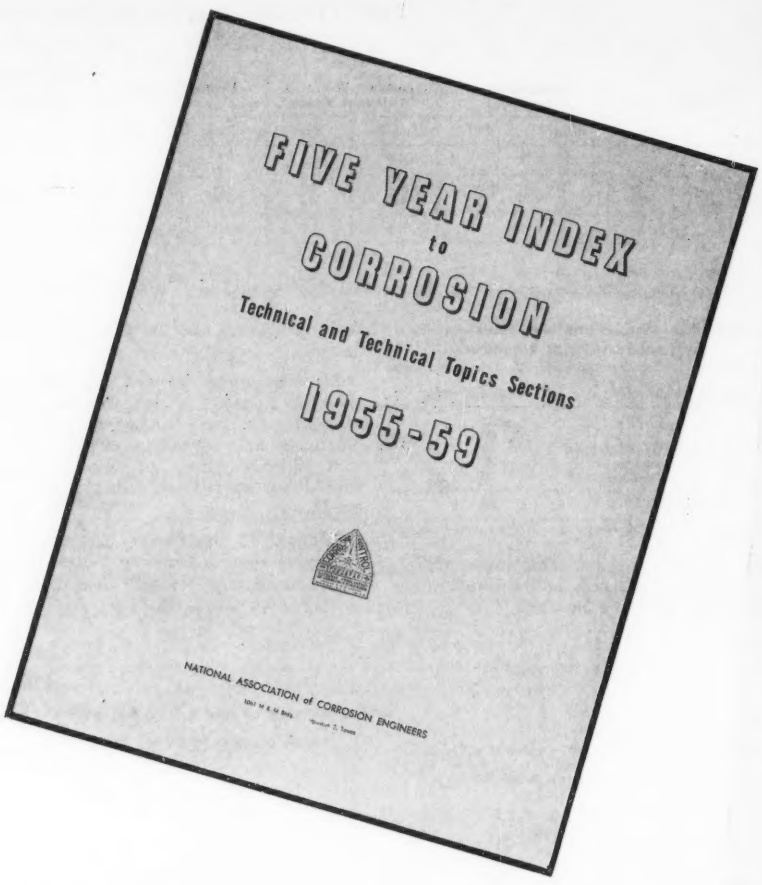

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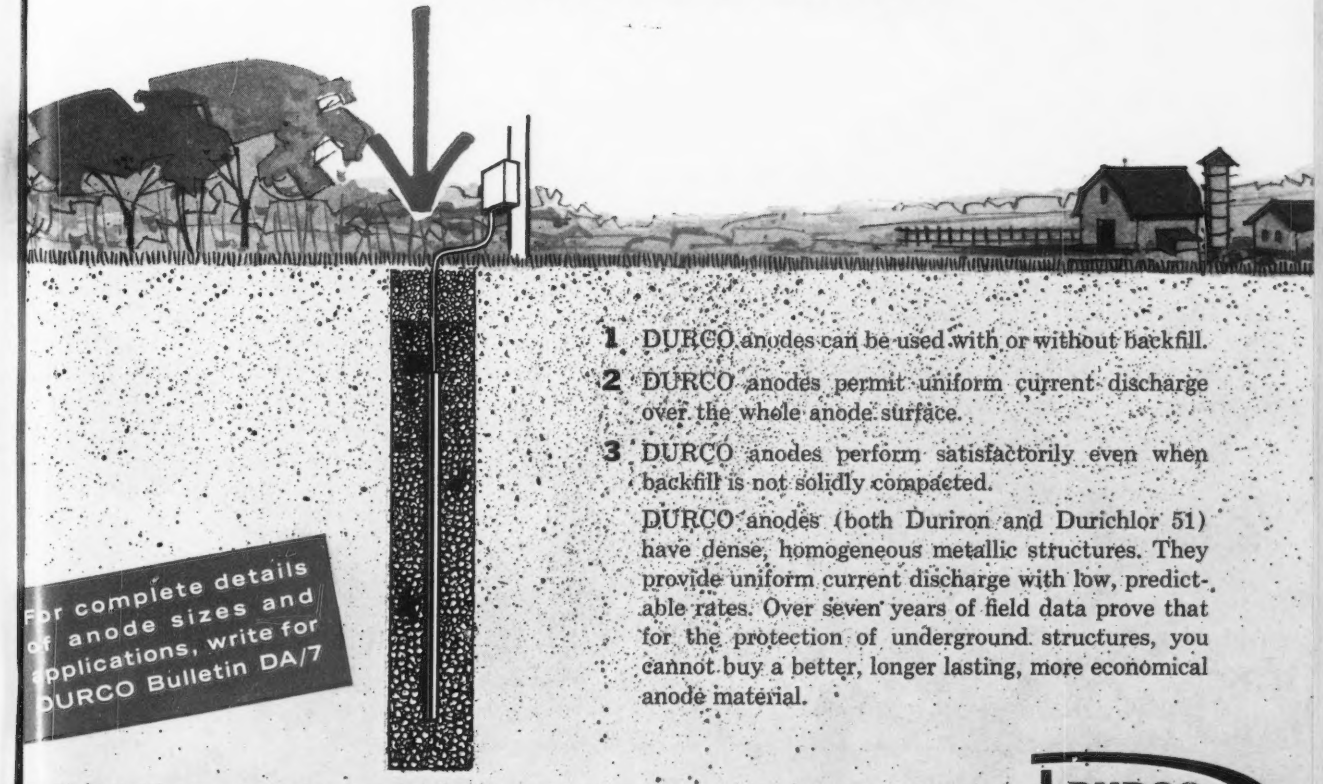

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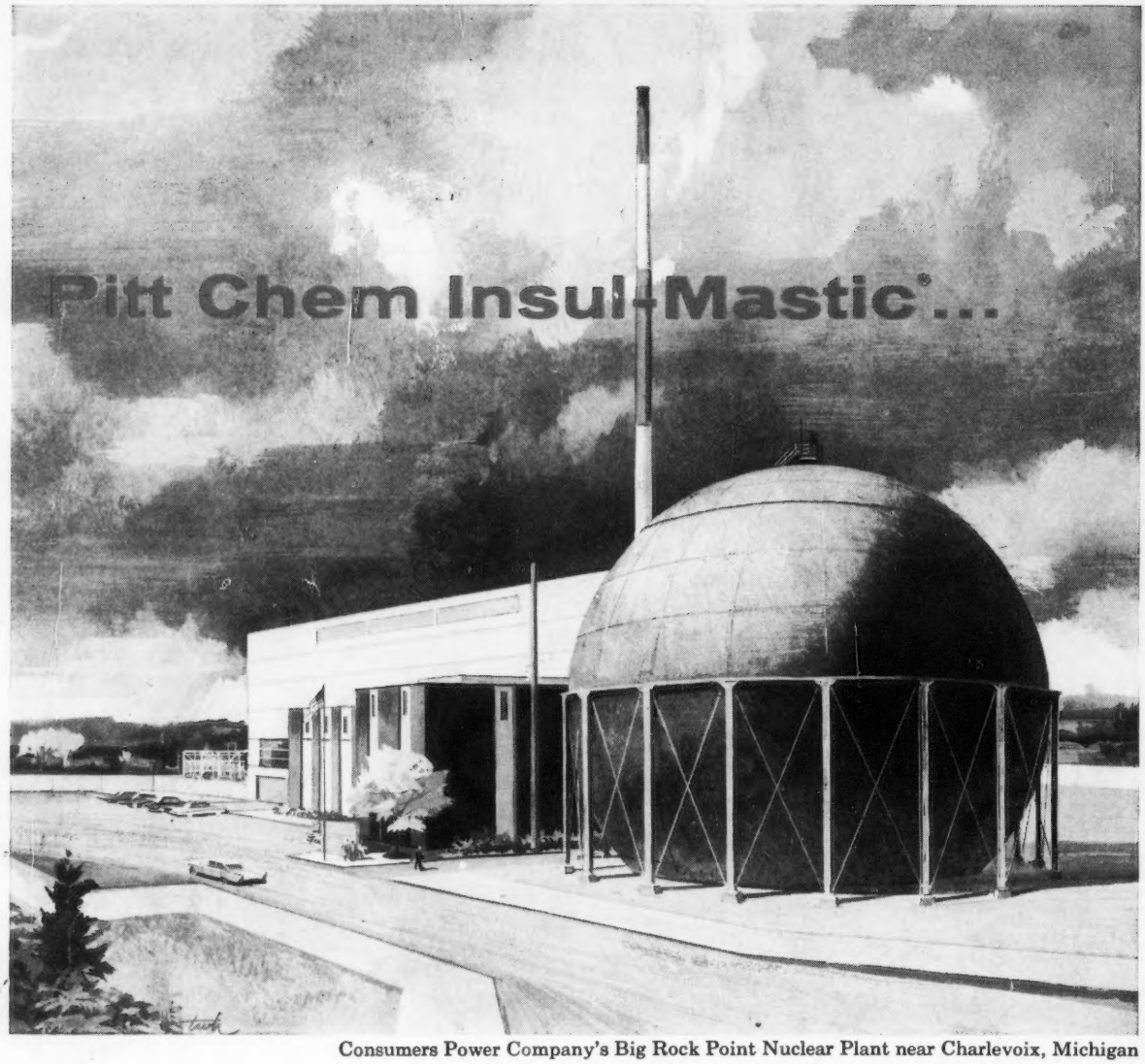

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